TETRA TECH

October 2, 2020

Mr. Robert Kondreck
Federal On-Scene Coordinator
U.S. Environmental Protection Agency, Region 5
77 West Jackson Boulevard
Chicago, Illinois 60604-3507

**Subject:** **Abbreviated Sampling and Analysis Plan (Revision 0)**
**Nelson Knitting Removal Assessment Site**
**Rockford, Winnebago County, Illinois**
**EPA Contract No. 68-HE-0519-D0005**
**Task Order - Task Order Line Item No. 68HE0520F0032-0001BI104**
**Document Tracking No. 0485**

Dear Mr. Kondreck:

Tetra Tech, Inc. (Tetra Tech) Superfund Technical Assessment and Response Team (START) is submitting, for your review and comment, the following abbreviated sampling and analysis plan (SAP) for the Nelson Knitting Removal Assessment Site located in Rockford, Winnebago County, Illinois. The SAP summarizes sample collection procedures that START will follow during the sampling event planned for October 8, 2020.

If you have any questions regarding this plan, please call me at (312) 201-7760.

Sincerely,

André Baker, Project Manager
Tetra Tech, Inc.

Enclosure
cc: Chris Burns, Tetra Tech START Program Manager
TOLIN file

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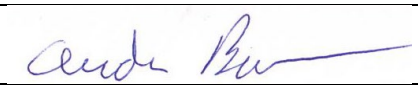
**ABBREVIATED SAMPLING AND ANALYSIS PLAN (REVISION 0)**
**FOR THE NELSON KNITTING REMOVAL ASSESSMENT SITE**

| | | |
|---|---|---|
| **TO-TOLIN#:** | F0032-0001BI104 | |
| **EPA OSC:** | Robert Kondreck | |
| **SITE NAME:** | Nelson Knitting Removal Assessment Site | |
| **SITE LOCATION:** | 909 South Main Street, Rockford, Illinois 61101 | |
| **SAMPLING ACTIVITIES:** | Asbestos, ambient air, paint chip, and waste sampling | |
| **SAMPLING DATES:** | October 8, 2020 | |
| **SAP PREPARER:** | André Baker | |
| **SIGNATURE/DATE** |  | 10/2/2020 |
| **QC REVIEWER:** | Brian Croft | |
| **SIGNATURE/DATE** |  | 10/2/2020 |
| **Document Tracking Number (DTN)** | 0485 | |

## SCOPE OF WORK AND OBJECTIVE:

The U.S. Environmental Protection Agency (EPA) Region 5 tasked Tetra Tech, Inc. (Tetra Tech) Superfund Technical Assessment and Response Team (START) with collecting environmental samples at the Nelson Knitting Removal Assessment Site (the site) under Task Order (TO) 68HE0520F0032 Task Order Line Item Number (TOLIN) 0001BI104 for EPA Contract number 68HE0519D0005. The overall goal of the sampling effort is to determine the risks posed to human health and the environment through their exposure to asbestos-containing material (ACM) and hazardous waste in the former Nelson Knitting facility.

To assess these risks, Tetra Tech and the U.S. EPA will perform the following activities during the removal assessment:

- Collect suspected bulk ACM to assess potential exposure due to material deterioration;
- Collect ambient air samples to assess the potential of asbestos releasing into the communities surrounding the site;
- Sample the liquid and/or solid waste located on site to assess the potential for a release to occur through the building floor drains and to assess the potential of exposure to workers and trespassers;
- Sample paint chips to assess the potential impact of deteriorating building debris; and
- Conduct real-time screening for mercury vapors and volatile organic compounds (VOC) to assess the potential exposure of workers and trespassers to these contaminants inside the building, and to evaluate if vandals/scrappers may have spread this contamination into the surrounding community.

The site, which is approximately two acres in size, contains a three-story manufacturing building that operated as a former sock knitting mill. The building has remained mostly vacant since 1990 but is frequented by trespassers. The approximate geographic coordinates of the facility are 42.263771 degrees

north and 89.101911 degrees west, as measured from the center of the property. The site is located in a mixed-use area of Rockford, Illinois, and is surrounded by commercial and vacant properties (Appendix A, Figures 1 and 2).

## SITE HISTORY:

The City of Rockford requested assistance with the Nelson Knitting site (the site) from the EPA Emergency Response Branch on June 29, 2020. The site, which is located at 909 South Main Street in Rockford, Illinois, operated as a manufacturing facility until approximately 1990. Since 1990, the site building has been vacant and is deteriorating.

During a Phase I Environmental Site Assessment update, conducted in 2009 for the City of Rockford, suspected ACM was observed on piping, steam pipe elbows, and on boilers within the building. Suspected lead paint was present throughout the building.

Other waste was also observed in the building, including containers of chemicals, mercury-containing fluorescent lamps, and suspected polychlorinated biphenyl (PCB)-containing fluorescent lamp ballasts and electrical capacitors. Sanborn maps indicated that the building's roof was composed of sheet rock and poured asbestos (FGA 2009).

On June 25, 2020, the City of Rockford determined that the entrance to the site was compromised. An inspection of the building found that the boilers and other steel products had been removed. Suspected ACM materials from the boilers and pipes were found discarded in piles on the floor, as well as in garbage bags and fiber drums. The roof was also observed to be failing in multiple locations.

On June 26, 2020, the City of Rockford condemned and secured the property (City of Rockford 2020).

## SAMPLING METHODS:

During the site assessment, Tetra Tech and EPA will collect potential bulk ACM, ambient air samples, and waste samples, and will screen the site for mercury, VOCs, and gamma radiation, as summarized below. Tetra Tech and EPA will:

- Collect up to 10 suspect bulk ACM samples (insulation, pipe wrap, etc.) at four locations that have been disturbed by activity in the building, including one duplicate sample for quality assurance/quality control (QA/QC), and analyze these samples for friable asbestos at a START-procured laboratory.
- Collect up to two ambient air samples for a minimum of 120 minutes (high-flow) at locations outside the facility and analyze these samples for asbestos at a START-procured laboratory.
- Collect up to eight ambient air samples (four high-flow and four low-flow) in high traffic or heavily damaged areas within the building and analyze the samples for asbestos at a START-procured laboratory.
- Collect up to six solid and/or liquid waste samples of hazardous materials that could be released to the floor drains, including one duplicate sample for QA/QC analysis, and analyze these samples for PCBs, flammability/combustibility, pH, and toxicity characteristic leaching procedure (TCLP) metals.
- Collect one wastewater sample from a floor sump (if accessible), including one duplicate sample for QA/QC analysis, and analyze this sample for PCBs, pH, flammability/combustibility,

semivolatile organic compounds (SVOC), VOCs, and total metals.

- Collect up to four paint chip samples from locations where paint is peeling and analyze these samples for lead and PCBs at a START-procured laboratory.
- Screen all areas of the building that appear to be structurally sound and any areas immediately outside the building where trespassing occurred. This screening will monitor for mercury vapors by using a real-time mercury vapor analyzer. During the mercury screening event, START will also screen for VOCs and gamma radiation by using a MultiRAE Pro five gas meter and a Ludlum model 44-9 detector, respectively.
- Photograph the sampling activities and sampling locations. Tetra Tech will record pertinent notes in the site logbook and include a general map of the sampling locations.
- Package and ship the samples to subcontracted laboratories (not yet identified).
- Submit a report that summarizes all field activities and validated analytical data and compares the data with background concentrations and appropriate pathway-specific benchmarks. Tetra Tech will also prepare a map with all sampling locations and field observations.

Tetra Tech will photograph the site and document activities in a logbook in accordance with Tetra Tech standard operating procedure (SOP) No. 024, "Recording Notes in Field Logbooks," and Tetra Tech's Quality Assurance Project Plan (QAPP) for START (Tetra Tech 2019). Sampling methods used to collect ACM samples, ambient air samples, paint samples, and waste samples (drum/container), and mercury-screening methods are provided in the sections below.

Figures are provided in Appendix A. Tables are shown in Appendix B; Table 1 is a compilation of total samples, including QC samples, and Table 2 is a compilation of analytical methods, sample volumes, containers, preservation techniques, and holding times. Appendix C contains the Tetra Tech SOPs that will be used during the site assessment. Attachment 1 contains non-Tetra Tech SOPs.

**Suspect Bulk ACM Sampling**

Bulk asbestos samples will be collected from four areas that have been disturbed by recent activity in the building. Asbestos samples that are easily assessable will be collected using the procedures outlined below.

The samples will be collected in Level C personal protective equipment (PPE). START will wet samples with amended water before placing the samples into labeled resealable plastic bags. START will identify specific sampling locations during the field activities, and the samples will be collected from areas with bulk asbestos piles. Approximately four building material samples will be collected. Samples will be shipped to a START-procured laboratory to be analyzed for asbestos fibers by EPA Test Method 600/R-93/116.

Disposable sampling equipment will be used at each sampling point to minimize the spread of asbestos fibers and cross-contamination.

**Ambient Air Sampling**

Over an 8-hour period, ambient air samples will be collected at up to two locations outside of the building and at up to four locations inside the building. Total air sampling time will be a minimum of 120 minutes. Tetra Tech will collect approximately 1,200 liters of air (high-volume samples) at an average flow rate of 10 liters per minute (L/min) and approximately 360 liters of air (low-volume samples) at an average flow rate of 3 L/min.

Each outdoor location will have a high-flow rate air pump (SKC QuickTake 30 pump or Gillian AirCon 2 air pump) operating at a flow rate of 10 L/min. Each indoor location will have both a high-flow rate air pump operating at a flow rate of 10 L/min and a low flow-rate air pump (GilAir Plus air pump) operating at a flow rate of 3 L/min. Each pump will have an attached 25-millimeter (mm)-diameter, 0.8-micrometer (µm) mixed cellulose ester membrane (MCE) filter cassette, mounted on a 4 to 5-foot tall tripod stand. The inlet cap of all filter cassettes will be removed (such that it is open-faced) during sampling, and the cassettes will be positioned downward. The flow rate of the air sampling train created from this assembly will be measured before and after samples are collected by using a Bios DryCal primary flow meter.

The detection limit for these samples will be set at 0.0012 fibers per cubic centimeter (f/cc) with a sensitivity of 0.0004 asbestos structures per cubic centimeter (s/cc). Sampling will be conducted in accordance with EPA/Environmental Response Team (ERT) SOP No. 2015, "Asbestos Sampling."

Samples will be analyzed for asbestos by National Institute for Occupational Safety and Health (NIOSH) Method 7400 through phased contrast microscopy (PCM). Results will be compared to a screening level of 0.05 f/cc, half the Occupational Safety and Health Administration (OSHA) Permissible Exposure Limit (PEL) for asbestos of 0.1 f/cc. Samples with a concentration of fibers above the screening level will be analyzed for asbestos by NIOSH Method 7402 through transmission electron microscopy (TEM).

**Waste Sampling**

Drums, containers, and the sump (if accessible) with liquids will be sampled in accordance with Tetra Tech SOP No. 008-2, "Containerized Liquid, Sludge, and Slurry Sampling." The drum and container liquid waste samples will be placed in a labeled, 8-ounce, clear, wide-mouth glass jar for laboratory analysis of PCBs, pH, and flammability/combustibility. The water from the sump will be analyzed for PCBs, pH, flammability/combustibility, SVOCs, VOCs, and total metals.

Solids will be collected by using dedicated equipment (i.e., disposable trowels) or a gloved hand. Up to six drum and container sampling locations will be selected based on observations obtained in the field and will be marked for future reference. All drum and container sampling will be conducted in accordance with the Tetra Tech health and safety plan (HASP). The drum and container solid waste samples will be placed in a labeled, 8-ounce, clear, wide-mouth glass jar for laboratory analysis of PCBs, pH, flammability/combustibility, and TCLP metals.

**Paint Chip Sampling**

Paint chip samples will be collected near the openings of the building where paint is peeling off the walls. The paint will be extracted by pulling on one end to create a chip. A minimum paint chip sample of two square inches will be collected. The color and location of the paint chip will be recorded in the field logbook and on the site figures. The paint chip samples will be placed inside a resealable plastic bag and will be submitted for lead and PCB analysis at a START-procured laboratory.

**Mercury, VOC, and Radiation Screening**

Mercury vapors will be screened in each structurally sound room and at each trespasser ingress and egress location by using a Jerome J505 mercury vapor analyzer. The Jerome mercury vapor analyzer can analyze mercury vapor at concentrations as low as 50 nanograms per cubic meter ($ng/m^3$).

A MultiRAE Pro will be used to screen for VOCs in each structurally sound room. The MultiRAE Pro is capable of detecting VOCs to 10 parts per billion (ppb).

A Ludlum model 44-9 detector will be used to screen for radiation in each structurally sound room. The Ludlum can detect gamma radiation at 3300 counts per minute per microrems per hour.

Tetra Tech will screen each room by first collecting measurements in the breathing zone. If the measurement is below the levels identified in the HASP, Tetra Tech will proceed with screening the rest of the room. The Jerome, Ludlum, and MultiRAE Pro will be moved across surfaces, in the breathing zone, or in areas that may contain contamination. If mercury, VOCs, or radiation is detected, the concentration will be recorded when the concentration ceases to increase and starts to decrease. No samples will be collected for laboratory analysis as part of the screening.

**SAMPLE NOMENCLATURE:**

Samples will be labeled according to a site-wide, generic nomenclature consisting of a site identifier (NK = Nelson Knitting.), sample description (Bulk = bulk asbestos, AA = ambient air, WW = waste water, LW = liquid waste, SW=solid waste, PC = paint chip), sample number, and date of collection. An example of a sample ID is "NK-LW-001-20201008" (a liquid waste sample, sample number 001, and date of collection on October 8, 2020). Sample locations will be marked in the field on a site map.

**SAMPLE HANDLING:**

The collected samples will be labeled, packaged, and shipped in accordance with procedures outlined in Tetra Tech's START QAPP (Tetra Tech 2019) and Tetra Tech SOP No. 019, "Packaging and Shipping Samples." The samples will be shipped under a signed chain-of-custody and will be analyzed at START-procured laboratories, yet to be determined.

**QUALITY ASSURANCE/QUALITY CONTROL:**

Field QA/QC measures for the asbestos samples will include the collection of one duplicate sample from each non-air sample per 10 samples. Tetra Tech will also collect two field blank samples and two matrix blank samples from each lot of MCE filter cassettes used.

Field QA/QC measures for the waste samples will include the collection of one duplicate sample per 10 samples. No matrix spike/matrix spike duplicate (MS/MSD) samples will be collected for the waste samples.

The Tetra Tech field team manager will be responsible for ensuring that sample quality and integrity are maintained, and sample label and documentation procedures are in accordance with the START QAPP and site-specific abbreviated SAP.

When the results are received, Tetra Tech START chemists will validate the laboratory results in accordance with Tetra Tech SOP No. 203, "Laboratory Analytical Data Verification – Minimum Requirements" and Response Engineering and Analytical Contract (REAC) SOP 1025 "Data Verification/Validation for Procedures for Asbestos in Air by TEM Analysis." Corrective actions may include resampling, reassessment of the laboratory's methods, and/or the addition of data qualifiers to the laboratory results.

## DECONTAMINATION:

Sampling equipment and PPE will be double-bagged and disposed of with all other used PPE waste produced at the site. Non-dedicated sampling equipment such as the air pumps will undergo a gross decontamination with deionized water in accordance with Tetra Tech SOP No. 002, "General Equipment Decontamination." A wet wipe may be used to decontaminate the air pumps. All investigation-derived waste (IDW) will be double-bagged by Tetra Tech and left on site. The IDW will be disposed of as dry industrial waste by the contractor performing the hazardous waste removal.

## SCHEDULE AND DELIVERABLES:

The sampling activities are scheduled to take place on October 8, 2020. Laboratory analytical results are anticipated to be available within two weeks following the receipt of the samples at the laboratory.

All laboratory analytical data from the samples will be validated by a Tetra Tech START chemist. The validated analytical results and other findings will be provided to EPA in the site assessment report. The anticipated schedule is outlined in the table below.

**PROPOSED SCHEDULE**

| Activities | Dates (Month, Day, Year) | | Deliverables | Deliverable Due Date |
|---|---|---|---|---|
| | Anticipated Date(s) of Initiation | Anticipated Date of Completion | | |
| HASP Preparation | September 24, 2020 | September 25, 2020 | HASP | October 5, 2020 |
| SAP Preparation | September 28, 2020 | October 5, 2020 | Abbreviated Sampling and Analysis Plan – Revision 0 | October 5, 2020 |
| Sample Collection | October 8, 2020 | October 8, 2020 | Logbooks, sampling and screening logs | Not applicable |
| Laboratory Analysis | October 9, 2020 | October 23, 2020 | Laboratory analytical report | Two (2) weeks after submittal of samples |
| Data Validation | October 26, 2020 | November 6, 2020 | Data validation report(s) | 10 business days after the receipt of the final laboratory analytical report(s) |
| Removal Assessment Report – Revision 0 | November 9, 2020 | November 23, 2020 | Removal Assessment Report – Revision 0 | Two (2) weeks after receiving the final data validation report(s) |
| Removal Assessment Report – Final Revision | Upon receipt of comments | One week after receipt of final client comments | Removal Assessment Report – Final Revision TBD | TBD |

**Notes:**
HASP Health and Safety Plan
SAP Sampling and Analysis Plan
TBD To be determined

**REFERENCES:**

City of Rockford. 2020. E-mail "Nelson Knitting – 909 S main St, Rockford, IL" From: Robert Wilhelmi, City of Rockford. To: Ramon Mendoza, EPA. June 29.

Fehr-Graham & Associates (FGA). 2009. Phase I Environmental Site Assessment Update and Phase II Environmental Site Assessment, Former Nelson Knitting Mills. July 9.

Tetra Tech, Inc. (Tetra Tech). 2019. Quality Assurance Project Plan (QAPP), Superfund Technical Assessment and Response Team (START V), Revision 1, U.S. Environmental Protection Agency Region 5, Solicitation No. 68HE0519D005. August.

# APPENDIX A

# SITE FIGURES


**Reference Map**


**Legend**

Approximate Site Boundary

Source: Modified from USGS, Illinois.
7.5-Minute (1:10,000 Scale) Topographic Map, 2018.
Rockford North Illinois. (1:10,000 Scale) Topographic Map, 2018.

Nelson Knitting Removal Assessment Site
909 South Main Street
Rockford, Winnebago County, Illinois

**Figure 1**

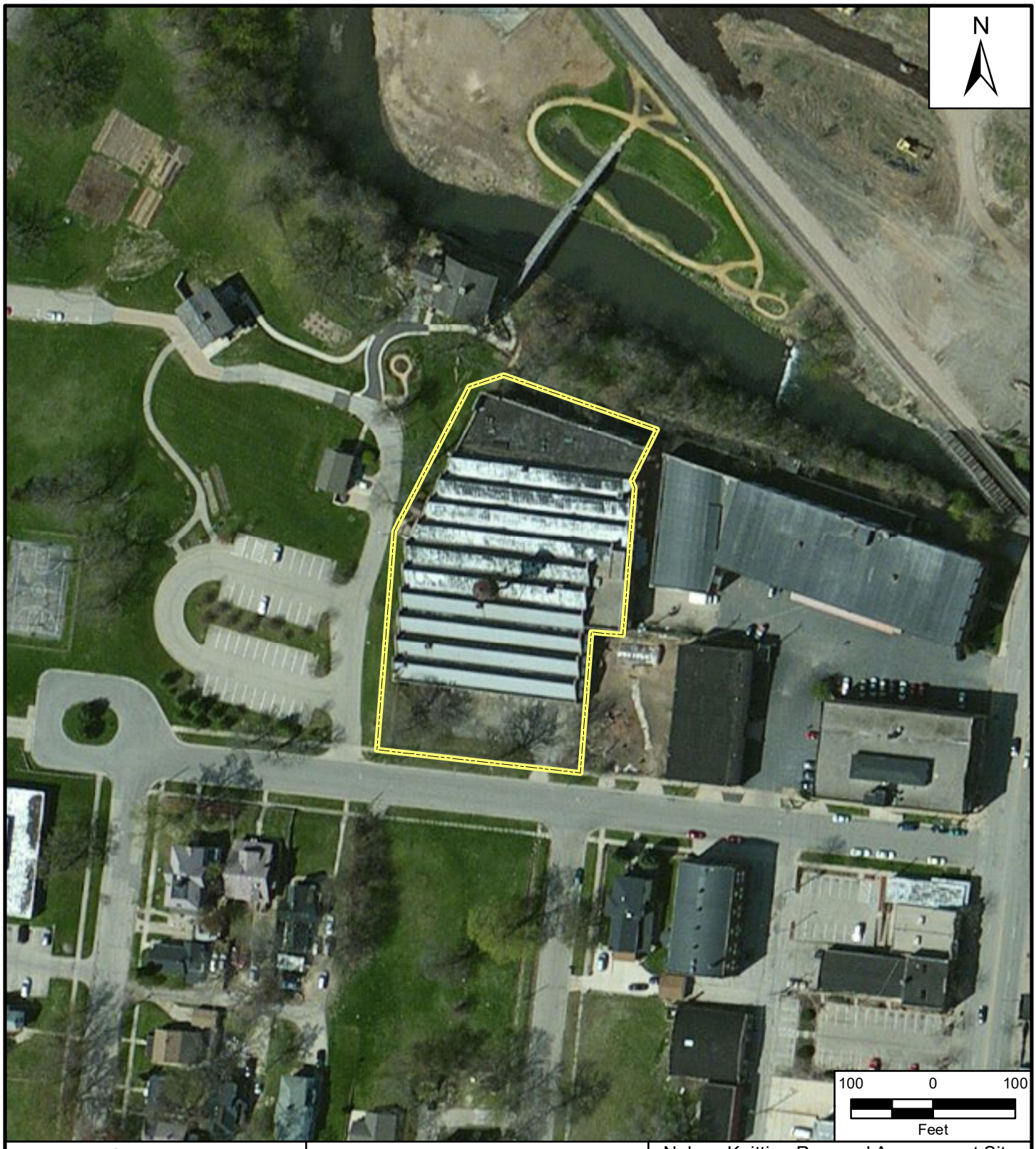
***Site Location Map***

TETRA TECH

| Prepared For: EPA | Prepared By: Tetra Tech, Inc. |
|---|---|

File Path: G:\IG\G9031-START V\Illinois\Nelson Knitting Site\mxd\Fig1_SiteLocation.mxd

Date Saved: 10/1/2020 EPA Contract No.: 68-HE-0519-D005 TO-TOLIN: F0032-0001BI104 Coordinate System: WGS 1984 Web Mercator Auxiliary Sphere Projection: Mercator Auxiliary Sphere Datum:WGS 1984 Units: Meter




**Reference Map**


**Legend**

Approximate Site Boundary

Nelson Knitting Removal Assessment Site
909 South Main Street
Rockford, Winnebago County, Illinois

**Figure 2**

***Site Layout Map***

TETRA TECH

| Prepared For: EPA | Prepared By: Tetra Tech, Inc. |
|---|---|

File Path: G:\G\G9031-START V\Illinois\Nelson Knitting Site\mxd\Fig2_SiteLayout.mxd

Date Saved: 10/1/2020 EPA Contract No.: 68-HE-0519-D005 TO-TOLIN: F0032-0001BI104 Coordinate System: WGS 1984 Web Mercator Auxiliary Sphere Projection: Mercator Auxiliary Sphere Datum:WGS 1984 Units: Meter

# APPENDIX B

# TABLES

**TABLE 1**
**SAMPLING REQUIREMENTS**

| Matrix | Parameter | Number of Investigative Samples[a] | Number of Quality Control (QC) Samples[b] | | | | | | Total Number of Investigative and QC Samples | Total Number of Sample Containers |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | Matrix Spike (MS) | Matrix Spike Duplicate (MSD) | Field Duplicate | Matrix Blank [c] | Field Blank | Trip Blank | | |
| Potential bulk ACM (pipe wrap, insulation, etc.) | Asbestos | 10 | 0 | 0 | 1 | 0 | 0 | 0 | 11 | 11 |
| Air (high-flow, outdoor ambient air) | Asbestos | 2 | 0 | 0 | 0 | 1 | 1 | 0 | 4 | 4 |
| Air (high- / low-flow indoor ambient air)[d] | Asbestos | 8 (4/4) | 0 | 0 | 0 | 1 | 1 | 0 | 10 | 10 |
| Paint Chips | Lead, PCBs | 4 | 0 | 0 | 0 | 0 | 0 | 0 | 4 | 4 |
| Waste (liquid and solid) | PCBs, pH, flammability/ combustibility, TCLP metals (solid only) | 6 | 0 | 0 | 1 | 0 | 0 | 0 | 7 | 7 |
| Waste (Sump Water) | PCBs, pH, flammability/ combustibility, metals, VOCs, SVOCs | 1 | 0 | 0 | 0 | 0 | 0 | 0 | 1 | 8 |

**Notes:**

[a] Refer to Table 2 for required sample volumes, containers, preservation techniques, and holding times.

[b] Refer to Worksheet 20 (Field Quality Control Sample Summary) of the Tetra Tech, Inc. START Region 5 Quality Assurance Project Plan (QAPP) for typical QC sample types and frequencies.

[c] Two matrix blanks will be collected to represent all asbestos air samples collected during the investigation.

[d] If a high-flow sample is overloaded, the laboratory will analyze the corresponding low-flow sample. Low-flow samples will only be analyzed if a high-flow sample is overloaded.

ACM Asbestos containing material

| | |
|---|---|
| EPA | U.S. Environmental Protection Agency |
| MS | Matrix Spike |
| MSD | Matrix Spike Duplicate |
| PCB | Polychlorinated biphenyls |
| QC | Quality Control |
| SVOC | Semivolatile organic compounds |
| TCLP | Toxicity characteristic leaching procedure |
| VOC | Volatile organic compounds |

**TABLE 2**
**SAMPLE VOLUMES, CONTAINERS, PRESERVATION TECHNIQUES, AND HOLDING TIMES**

| Matrix | Parameter | Method of Analysis | Volume and Container | Preservation Techniques | Holding Time (Extraction/Analysis) |
|---|---|---|---|---|---|
| Potential bulk ACM | Asbestos | EPA Test Method 600/R-93/116 | Sealable plastic bag | NA | NA |
| Air (Ambient) | Asbestos | NIOSH 7400 and 7402 | Minimum of 360 L (low-flow samples) and 1200 L (high-flow samples) in 25-mm-diameter, 0.8- µm MCE filter cassette | NA | NA |
| Paint Chips | Lead | SW-846 6010C | Zip-tight baggie | Store at 4 ºC | 6 months |
| Paint Chips | PCBs | SW-846 8082A | Zip-tight baggie | Store at 4 ºC | 14 days to extract<br>40 days to analyze extract |
| Waste (liquid and solid) | PCBs | SW-846 8082A | One 8-ounce glass jar with Teflon lined cap | Store at 4 ºC | 14 days to extract<br>40 days to analyze extract |
| | pH | SW-846 9040C/9045D | | | 7 days |
| | Flammability / Combustible | SW-846 1010 | | | NA |
| | TCLP Metals | SW-846 8260B | | | 6 months (28 days for mercury) |
| Waste (Sump Water) | SVOCs | SW-846 8270C | Two 1-L amber glass bottles fitted with PTFE lined screw caps | Store at 4 ºC | 7 days to extract<br>40 days to analyze extract |
| | VOCs | SW-846 8260B | Three 40-mL VOA vials with Teflon-lined septum | HCl to pH<2; store at 4 ºC | 14 days to extract<br>14 days to analyze extract |
| | PCBs | SW-846 8082A | Two 1-L amber glass bottles fitted with PTFE lined screw caps | Store at 4 ºC | 7 days to extract<br>40 days to analyze extract |
| | Total Metals | SW-846 6010C | One 1-L HDPE Poly Bottle | $HNO_3$ / Store at 4 ºC | 28 days |
| | pH | SW-846 9040C | 250-mL glass jar | Store at 4 ºC | 24 hours |
| | Flammability / Combustible | SW-846 1010 | 250-mL HDPE bottle | None | NA |

**Notes:**

| | |
|---|---|
| ACM | Asbestos containing materials |
| EPA | U.S. Environmental Protection Agency |
| HCL | Hydrochloric acid |
| HDPE | High-density polyethylene |
| $HNO_3$ | Nitric acid |
| ISO | International Standards Organization |
| L | Liter |
| ºC | Degrees Celsius |
| µm | Micrometer |

| | |
|---|---|
| MCE | Mixed Cellulose Ester |
| mL | Milliliter |
| mm | Millimeter |
| NIOSH | National Institute for Occupational Safety and Health |
| NA | Not applicable |
| PCB | Polychlorinated biphenyls |
| PCME | Phase Contrast Microscopy Equivalent |
| PTFE | Polytetrafluoroethylene (Teflon) |
| SW-846 | U.S. Environmental Protection Agency, Test Methods for Evaluating Solid Waste, Physical/Chemical Methods (SW-846) available at the following web address: http://www.epa.gov/epawaste/hazard/testmethods/sw846/online/index.htm. |
| SVOC | Semivolatile organic compound |
| TCLP | Toxicity characteristic leaching procedure |
| TEM | Transmission electron microscopy |
| VOA | Volatile organic analysis |
| VOC | Volatile organic compound |

## APPENDIX C

## TETRA TECH (TT)
## STANDARD OPERATING PROCEDURES (SOP)

| SOP NO. | TITLE |
|---|---|
| TT SOP No. 002 | General Equipment Decontamination |
| TT SOP No. 008-2 | Containerized Liquid, Sludge, and Slurry Sampling |
| TT SOP No. 019 | Packaging and Shipping Samples |
| TT SOP No. 024 | Recording Notes in Field Logbooks |
| TT SOP No. 203 | Laboratory Analytical Data Verification – Minimum Requirements |

**SOP APPROVAL FORM**

TETRA TECH EM INC.

ENVIRONMENTAL STANDARD OPERATING PROCEDURE

# GENERAL EQUIPMENT DECONTAMINATION

**SOP NO. 002**

**REVISION NO. 4**

Last Reviewed: March 2018

Quality Assurance Approved

March 9, 2018

Date

# 1.0 BACKGROUND

All nondisposable field equipment must be decontaminated before and after each use at each sampling location to obtain representative samples and to reduce the possibility of cross-contamination.

## 1.1 PURPOSE

This standard operating procedure (SOP) establishes the requirements and procedures for decontaminating equipment in the field.

## 1.2 SCOPE

This SOP applies to decontaminating general nondisposable field equipment. All sampling equipment must be thoroughly cleaned before each use to prevent contamination of samples.

## 1.3 DEFINITIONS

**Alconox:** Phosphate-containing soap, obtained in powder form and dissolved in water

**Liquinox:** Phosphate-free soap, obtained in liquid form for mixing with water

## 1.4 REFERENCES

U.S. Environmental Protection Agency (EPA). 1992a. "Guide to Management of Investigation-Derived Wastes." Office of Solid Waste and Emergency Response. Washington, DC. EPA 9345.3-03FS. January.

EPA. 1992b. "RCRA Ground-Water Monitoring: Draft Technical Guidance." Office of Solid Waste. Washington, DC. EPA/530-R-93-001. November.

EPA. 2015. "Field Equipment Cleaning and Decontamination." Science and Ecosystem Support Division SESDPROC-205-R3 (Rev. 3, 12/18/15). https://www.epa.gov/quality/field-equipment-cleaning-and-decontamination

## 1.5 REQUIREMENTS AND RESOURCES

The equipment and supplies to conduct decontamination may include the following:

- Scrub brushes
- Large wash tubs or buckets
- Squirt bottles
- Alconox or Liquinox (Note: Alconox contains phosphates, and phosphates have been banned in many household cleaning products based on their adverse effect on the environment.)
- Tap water
- Distilled water
- Deionized water
- Plastic sheeting
- Aluminum foil
- Isopropanol (laboratory grade)

# 2.0 PROCEDURE

The procedures below discuss decontamination of personal protective equipment (PPE) as well as equipment for drilling and monitoring well installation, borehole soil sampling, general sampling, water level measurement, and groundwater sampling. PPE as outlined in the site-specific health and safety plan should be used during decontamination procedures. Special handling of used PPE and wastewater generated from decontamination procedures may be required if the type of contamination is considered hazardous according to the Resource Conservation and Recovery Act (RCRA). Any special handling should also be outlined in the site-specific health and safety plan or the sampling and analysis plan.

Some clients may have additional requirements for decontamination procedures. For example, phosphate-free detergent may be a requirement and, therefore, it would not be appropriate to use Alconox.

Source water for decontamination should be selected based on site-specific conditions and contaminants. Organic-free water would be more appropriate to use at sites where organic compounds are being investigated; conversely, laboratory-grade deionized water would be more appropriate where low levels of contaminants are being investigated. Standard distilled water, readily available at grocery stores, may be appropriate at other times. Refer to the site-specific sampling and analysis plan for details concerning source water.

In general, conduct field activities to move from cleaner to more contaminated locations to minimize the potential for cross contamination between locations.

## 2.1 PERSONAL PROTECTIVE EQUIPMENT DECONTAMINATION

Personnel working in the field are required to follow specific procedures for decontamination prior to leaving the work area so that contamination is not spread off site or to clean areas. Refer to the site-specific health and safety plan as the first resource for types of PPE; not all types of PPE nor methods for decontamination discussed below will be appropriate for every site. All used disposable protective clothing, such as Tyvek, coveralls, gloves, and booties, will be containerized for later disposal. Decontamination water will be containerized in 55-gallon drums (refer to Section 3.0).

Personnel decontamination procedures will be as follows:

1. Select an area removed from sampling locations that is both downwind and downgradient. Decontamination must not cause cross-contamination between sampling points.

2. Wash neoprene boots (or neoprene boots with disposable booties) with Liquinox or Alconox solution and rinse with clean water. Remove booties and retain boots for subsequent reuse.

3. Remove outer gloves and place into plastic bag for disposal.

4. Remove Tyvek or coveralls. Containerize Tyvek for disposal and place coveralls in plastic bag for reuse.

5. Remove air purifying respirator (APR), if used, and place the spent filters in a plastic bag for disposal. Filters should be changed daily or sooner, depending on use and application. Place the respirator into a separate plastic bag after it has been cleaned and disinfected according to the instructions for the respirator.

6. Remove disposable gloves and place them in plastic bag for disposal.

7. Thoroughly wash hands and face in clean water and soap.

## 2.2 DRILLING AND MONITORING WELL INSTALLATION EQUIPMENT DECONTAMINATION

All drilling equipment should be decontaminated at a designated location on site before drilling operations begin, between borings, and at completion of the project. Decontamination may be conducted on a temporary decontamination pad constructed at a satellite location within the site. The purpose of the decontamination pad is to contain wash waters and potentially contaminated soil generated during decontamination procedures. Decontamination pads may be constructed of concrete, wood, or plastic sheeting, depending on the site-specific needs and plans. Wash waters and contaminated soil generated during decontamination should be considered investigation-derived waste (IDW) and, thus, should be collected and containerized for proper disposal.

Monitoring well casing, screens, and fittings are assumed to be delivered to the site in a clean condition. However, they may be steam cleaned and placed on polyethylene sheeting on site before they are used downhole, if required by the site-specific work plan. The drilling subcontractor will typically furnish the steam cleaner and water.

The drilling auger, bits, drill pipe, any portion of drill rig that is over the borehole, temporary casing, surface casing, and other equipment used in or near the borehole should be decontaminated by the drilling subcontractor as follows:

1. Select an area removed from sampling locations that is both downwind and downgradient. Decontamination must not cause cross-contamination between sampling points.
2. Remove loose soil using shovels, scrapers, wire brushes, and any related material.
3. Steam clean or pressure wash to remove all visible dirt. Use appropriate PPE (for example, a face shield and Tyvek/coveralls) as necessary.
4. If equipment has directly or indirectly contacted contaminated media and is known or suspected of being contaminated with oil, grease, polycyclic aromatic hydrocarbons (PAH), polychlorinated biphenyls (PCB), or other hard-to-remove organic materials, rinse equipment with laboratory-grade isopropanol.
5. To the extent possible, allow components to air dry; drying helps limit the spread of contamination through contact.
6. All wastewater from decontamination procedures should be containerized.

## 2.3 BOREHOLE SOIL SAMPLING DOWNHOLE EQUIPMENT DECONTAMINATION AND GENERAL SOIL SAMPLING EQUIPMENT DECONTAMINATION

All soil sampling equipment should be decontaminated before use and after each sample as follows:

1. Select an area removed from sampling locations that is both downwind and downgradient. Decontamination must not cause cross-contamination between sampling points.

2. Scrub the split-barrel sampler and sampling tools in a wash bucket or tub using a stiff, long-bristle brush with a solution of tap water with Liquinox or Alconox.

3. Rinse equipment thoroughly with tap water or distilled water.

4. Perform a final rinse with deionized or distilled water. Refer to the site-specific sampling and analysis plan for requirements for deionized or distilled water.

5. Place cleaned equipment in a clean area on plastic sheeting or aluminum foil and allow to air-dry.

6. Containerize all water and rinsate; disposable single-use sampling equipment should also be containerized.

## 2.4 WATER LEVEL MEASUREMENT EQUIPMENT DECONTAMINATION

Field personnel should decontaminate the well sounder and interface probe before inserting and after removing them from each well. The following decontamination procedures should be used:

1. Select an area removed from sampling locations that is both downwind and downgradient. Decontamination must not cause cross-contamination between sampling points.

2. Wipe the tape and probe with a disposable Alconox- or Liquinox-impregnated cloth or paper towel.

3. If immiscible layers are encountered, the interface probe may require steam cleaning or washing with laboratory-grade isopropanol.

4. Rinse with distilled or deionized water.

5. Containerize all water and rinsate for proper disposal.

## 2.5 GROUNDWATER SAMPLING EQUIPMENT

The following procedures are to be employed to decontaminate equipment used for groundwater sampling. Decontamination is not necessary when using disposable (single-use) pump tubing or bailers. Bailer and downhole pumps decontamination procedures are described in the following sections.

### 2.5.1 Bailers

1. Select an area removed from sampling locations that is both downwind and downgradient. Decontamination must not cause cross-contamination between sampling points.

2. Remove and containerize any purge water remaining in the bailer.

3. Scrub the inside and outside of the bailer in a wash bucket or tub using a stiff, long-bristle brush with a solution of tap water with Liquinox or Alconox. Select cleaning equipment that will not scratch or damage the bailer.

4. Rinse the bailer thoroughly with tap water or distilled water.

5. If groundwater contains or is suspected to contain oil, grease, PAHs, PCBs, or other hard-to-remove organic materials, rinse equipment with laboratory-grade isopropanol.

6. Perform a final rinse with deionized or distilled water.

7. Allow the cleaned bailer to air dry.

8. Wrap the bailer in aluminum foil or a clean plastic bag for storage.

9. Containerize the decontamination wash waters for proper disposal.

### 2.5.2 Downhole Pumps

1. Select an area removed from sampling locations that is both downwind and downgradient. Decontamination must not cause cross-contamination between sampling points.

2. Remove and containerize any purge water in the pump and tubing and dispose of tubing.

3. Dismantle the pump as much as possible and scrub components in a wash bucket or tub using a stiff brushes of appropriate size with a solution of tap water with Liquinox or Alconox.

4. Rinse pump components thoroughly with tap water or distilled water.

5. If groundwater contains or is suspected to contain oil, grease, PAHs, PCBs, or other hard-to-remove organic materials, rinse the pump and tubing with laboratory-grade isopropanol.

6. Perform a final rinse with deionized or distilled water.

7. Allow components to air dry.

8. Wrap pump in aluminum foil or a clean plastic bag for storage.

9. Containerize the used tubing and decontamination wash waters for proper disposal.

## 3.0 INVESTIGATION-DERIVED WASTE

IDW can include disposable single-use PPE and sampling equipment, soil cuttings, and decontamination wash waters and sediments. Requirements for waste storage may differ from one facility to the next. Facility-specific directions for waste storage will be provided in project-specific documents, or separate direction will be provided by the project manager. The following guidelines are provided for general use:

1. Assume that all IDW generated from decontamination contains the hazardous chemicals associated with the site unless there are analytical or other data to the contrary. Waste solution volumes could vary from a few gallons to several hundred gallons in cases where large equipment required cleaning.

2. Containerized waste rinse solutions are best stored in 55-gallon drums (or equivalent containers) that can be sealed until ultimate disposal at an approved facility.

3. Label IDW storage containers with the facility name and address, date, contents, company generating the waste, and an emergency contact name and phone number.

4. Temporarily store the IDW in a protected area that provides access to the containers and allows for spill/leak monitoring, sampling of containers, and removal after the disposal method has been identified.

**SOP APPROVAL FORM**

TETRA TECH, INC.

EMI OPERATING UNIT

ENVIRONMENTAL STANDARD OPERATING PROCEDURE

# CONTAINERIZED LIQUID, SLUDGE, AND SLURRY SAMPLING

**SOP NO. 008**

**REVISION NO. 3**

Last Reviewed: August 2020

Carla Jarvis

Quality Assurance Approved

*August 11, 2020*

Date

# 1.0 BACKGROUND

Taking samples of liquid, sludge, and slurry from drums or other containers can present some unique problems. Manmade containers are typically closed. Containers are usually accessed either through small ports, manways, hatches, taps, or bungs. The size, shape, construction material, and location of a container may limit the types of equipment and methods that can be used to collect samples.

## 1.1 PURPOSE

This standard operating procedure (SOP) establishes procedures for sampling liquid, sludge, and slurry from containers.

## 1.2 SCOPE

Opening a closed container is a potentially hazardous task because toxic vapors and/or explosive gases could potentially be released. Whenever containers that may contain hazardous materials are to be opened for sampling or any other reason, the sampling team should follow appropriate guidelines provided in site-specific sampling plans, health and safety plans, and the general guidelines in this SOP.

How containers are opened will depend on (1) the purpose of the sampling; (2) site conditions; (3) the number, type, and condition of the containers; and (4) the anticipated type of media to be sampled. As a result, no comprehensive procedures can be defined for sampling all types of containerized liquid, sludge, and slurry. This SOP provides general guidelines for dealing with problems that may be encountered while opening containers and sampling the media. General procedures are provided for sampling containerized liquid, sludge, and slurry using glass tubes and composite liquid waste samplers (COLIWASA).

## 1.3 DEFINITIONS

**Bung Remover:** A device used to open the lid of a drum.

**COLIWASA:** Composite liquid waste sampler used to sample free-flowing liquids and slurries in containers.

**Hazardous Samples:** Hazardous samples include dangerous goods and hazardous substances.

Hazardous samples shipped by air should be packaged and labeled in accordance with procedures specified by the International Air Transportation Association (IATA) Dangerous Goods Regulations (DGR); ground shipments should be packaged and labeled in accordance with the U.S. Department of Transportation (DOT) Hazardous Materials Regulations (HMR, *Code of Federal Regulations*, Title 49 [49 CFR] Parts 106 through 180). See SOP No. 019 (Packaging and Shipping Samples) for additional information regarding training and directions for personnel managing or shipping hazardous waste samples.

**Photoionization Detector (PID):** A direct-reading air monitoring instrument used to measure organic vapors based on the principle of photoionization. Examples of PIDs include the RAE Systems MiniRAE 3000 and the Ion Science PhoCheck TIGER.

**Flame Ionization Detector (FID):** A direct-reading, air monitoring instrument used to measure organic vapors based on the principle of flame ionization. An example of an FID is ThermoFisher Scientific TVA2020.

## 1.4 REFERENCES

American Society for Testing and Materials (ASTM). 2016. "Standard Practice for Sampling with a Composite Liquid Waste Sampler (COLIWASA)." ASTM D5495-03(2016). September 1.

ASTM. 2018. "Standard Guide for Sampling of Drums and Similar Containers by Field Personnel." ASTM D 6063-11(2018).

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## 1.5 REQUIREMENTS AND RESOURCES

Depending on container specifications and the method selected for collecting samples, the following equipment may be required to sample liquid, sludge, and slurry from containers:

- Glass tubes
- FID or PID

- Bung remover
- COLIWASA
- Rubber stopper
- Stainless-steel spatula
- Chain-of-custody forms and shipping materials
- Sample containers and labels
- Appropriate personal protective equipment (PPE)
- A permanent marker for labeling containers

## 2.0 PROCEDURES

Opening a closed container may potentially release toxic vapors and/or explosive gases. The decision to open a container to sample the contents should be made only after other potentially less dangerous site characterization methods, such as geophysical investigations or sampling of noncontainerized media, have been ruled out. In some cases, however, sampling the contents of the container may be necessary for use in legal cases or for other reasons.

Until the container contents are characterized, the sampling team should assume that materials in unlabeled containers are hazardous. Labeled containers (such as 55-gallon drums) are often reused and can be mislabeled. The sampling team should exercise caution when working with or around containers.

When the decision to open a container has been made, the sampling team must assess potential exposure risks. Risk factors include the number, type, and condition of containers; site conditions that could prevent a container from being safely and efficiently opened; and the anticipated contents of the container. Based on this information and based on the scope of work for the project, the sampling team should consist of at least two persons and develop a safe procedure for opening the container and sampling its contents.

Sampling team members must wear appropriate PPE when opening and sampling containers. In some cases, particularly when the contents of the container are not positively known, the sampling team should consider using a remote drum opener to open closed containers. The choice of remote drum opening methods depends on the number of drums to be opened, their contents, and their physical condition. One type of remote drum opener uses hydraulic pressure to push a non-sparking metal spike into either the side or top of the drum.

After the container is opened, headspace gases should be monitored using an intrinsically safe monitoring instrument. At a minimum, a preliminary check using appropriate air-monitoring instruments should be conducted to help determine the level of PPE required and the appropriate sampling method.

Layering or stratification of any material left undisturbed over time is likely. Agitation of the container to homogenize the material can be difficult and is undesirable if the contents of the container are not known. The sampling team must ensure that samples represent the entire contents of the container, not just the contents of a single layer.

For sampling liquid and sludge in drums or other small to medium-sized containers, the glass tube sampling method is recommended. Tubes are available that collect a sample from the full depth of a drum and retain it until placement in a sample container. This sampling method is discussed in detail in Section 2.1. The COLIWASA is widely used to sample containerized and free-flowing liquids and slurries in drums and other containers. It also is used for sampling immiscible liquid-phase waste. Use of the COLIWASA is outlined in Section 2.2.

## 2.1 SAMPLING USING GLASS TUBES

Glass tubes can be used to sample liquids and sludge in containers such as 55-gallon drums. Glass tubes designed for this purpose are normally 122 centimeters (4 feet) long and have an inside diameter of 0.6 to 1.6 centimeters (0.24 to 0.63 inches). Glass tubes with larger inside diameters are used for sampling viscous liquids. When sampling is completed, the glass tubes can be broken and disposed of in the container just sampled. This eliminates the need for cleanup and disposal. However, if disposal of the tube by breaking in into the drum interferes with plans for the removal of the container contents, other disposal techniques should be evaluated.

The glass tube method is a quick, relatively inexpensive way of sampling containerized liquid and sludge. The major disadvantage of this method is that some sample loss may occur when sampling less viscous liquids. Splashing of such liquids also can expose sampling team members to potentially hazardous materials. For this reason, appropriate PPE, such as a butyl rubber apron, a face shield, safety glasses, respirators, boot covers, and gloves may be needed when using the glass tube method.

The procedures for sampling liquids and sludge using the glass tube method are given below. Following these procedures, cautionary notes are provided.

### 2.1.1 Sampling Containerized Liquids Using a Glass Tube

The following procedures can be used to sample containerized liquids using a glass tube:

1. Place all sampling equipment on a plastic sheet next to the container to be sampled. Sample containers should be selected in accordance with the requirements identified in the site field sampling plan or Quality Assurance Project Plan.

2. Affix a completed sample container label to the appropriate sample container.

3. Wear appropriate PPE. Use a PID or FID to monitor airborne organic vapors and gases in the breathing zone. In most cases, a PID is preferred because it is intrinsically safe, although an FID may be appropriate in some cases.

4. Record in the field logbook all exterior container markings, special conditions, and the type of opening through which the sample will be collected.

5. Using a permanent marker, make a unique identifying number on the container.

6. Locate an existing opening or bung hole in the container, if possible.

7. Using non-sparking tools, a bung remover, or a remote drum opener, carefully remove the cover or bung from the container.

8. Slowly insert a glass tube to a level slightly above the bottom of the container or until a solid layer is encountered. If layering or stratification of the liquids in the container is expected, the glass tube should be inserted at a rate that permits the liquid level inside and outside the tube to be about the same. Keep at least 30 centimeters (12 inches) of the glass tube above the top of the container.

9. Allow the liquid in the container to reach its natural level in the glass tube.

10. Cap the top of the glass tube with a safety-gloved thumb or a rubber stopper.

11. Remove the capped glass tube from the container, look for different layers, and insert the uncapped end into the labeled sample container.

12. Release the thumb or rubber stopper from the glass tube to allow the liquid to drain into the sample container.

13. Fill the sample container to approximately 90 percent of its capacity. Repeat steps 8 through 12 if more volume is needed to fill the sample container.

14. Dispose of the glass tube in an appropriate manner.

15. Ensure that a Teflon® liner is present in the sample container cap. Secure the cap tightly on the sample container. All containerized liquid samples should be evaluated in accordance with the “Sample Classification” section of SOP No. 019 (Packaging and

Shipping Samples) to determine if they are hazardous samples; hazardous samples should be packaged and shipped in accordance with Dangerous Goods Regulations.

16. Replace the bung in the container or seal the opening in the container with plastic.

17. Complete all chain-of-custody forms and record sampling activities in the field logbook or on field data forms. Unless the sample will be analyzed at the site, complete all sample packaging requirements in accordance with SOP No. 019, Packaging and Shipping Samples.

### 2.1.2 Sampling Containerized Sludge Using a Glass Tube

The following procedures can be used to sample containerized sludge using a glass tube.

1. Follow steps 1 through 7 for sampling containerized liquids using a glass tube (see Section 2.1.1).

2. Slowly insert a glass tube to a level slightly above the top of the sludge layer. Keep at least 30 centimeters (12 inches) of the glass tube above the top of the container.

3. Allow the liquid in the container to reach its natural level in the glass tube.

4. Gently push the glass tube into the sludge layer at the bottom of the container.

5. Cap the top of the glass tube with a safety-gloved thumb or a rubber stopper.

6. Remove the capped glass tube from the container and insert the uncapped end into the labeled sample container (for example, a wide-mouthed, 8-ounce glass jar).

7. Release the thumb or rubber stopper from the glass tube to allow the material to drain into the sample container. If necessary, the sludge sample in the bottom of the tube may be dislodged using a stainless-steel spatula.

8. Fill the container to approximately 90 percent of its capacity. Repeat steps 2 through 7 if more volume is needed to fill the sample container.

9. Dispose of the glass tube in an appropriate manner.

10. Ensure that a Teflon® liner is present in the sample container cap. Secure the cap tightly on the sample container. All containerized sludge samples should be evaluated in accordance with the “Sample Classification” section of SOP No. 019 (Packaging and Shipping Samples) to determine if they are hazardous samples; hazardous samples should be packaged and shipped in accordance with Dangerous Goods Regulations.

11. Replace the bung in the container or seal the opening in the container with plastic.

12. Complete all chain-of-custody forms and record sampling activities in field logbook and any field data forms. Unless the sample will be analyzed at the site, complete all sample packaging requirements in accordance with SOP No. 019, Packaging and Shipping Samples.

### 2.1.3 Cautionary Notes

Because there is potential for problems, interferences, and accidents to occur during sampling of containerized liquids and sludges, the following cautionary notes are provided.

1. If you observe any reaction when the glass tube is inserted into the container (for example, violent agitation, smoke, light, or heat), leave the area immediately.

2. If the glass tube becomes cloudy or smoky after inserting it into the container, hydrofluoric acid may be present. Glass tube sampling is inappropriate in this circumstance. Instead, use a comparable length of rigid plastic tubing to collect the sample and transfer the sample to an appropriate sample container.

3. When solid material is encountered in a container (either a floating layer or bottom sludge), use the sludge sampling procedure to collect a sample of the material. Alternatively, if the container opening is sufficiently large, the material may be sampled with a disposable scoop attached to a disposable wooden or plastic rod.

## 2.2 SAMPLING USING THE COLIWASA

The COLIWASA is used to collect samples of containerized or free-flowing liquid and slurry in drums and other containers. The COLIWASA is commercially available; however, it can be assembled from a variety of materials, including polyvinyl chloride (PVC), glass, or Teflon®. It consists of a 152-centimeter (5-foot) -long tube with an inside diameter of 4 centimeters (1.6 inches). The tube has a neoprene stopper at one end attached by a rod running the length of the tube to a locking mechanism at the other end. Manipulation of the locking mechanism opens and closes the COLIWASA by raising and lowering the neoprene stopper.

The recommended COLIWASA design is shown in Figure 1. The design may be modified to meet the needs of a sampling situation. The major drawbacks of using a COLIWASA involve decontamination and cost. The COLIWASA is difficult to decontaminate in the field and has a high cost compared to glass tubes. However, disposable COLIWASAs are available and are a viable alternative. The COLIWASA's major advantage is its ability to collect samples that accurately represent a multiphase waste.

The following procedure can be used for sampling containerized liquid or slurry using the COLIWASA:

1. If a commercial COLIWASA is unavailable, select the material to make the COLIWASA (for example, PVC, glass, or Teflon®). Assemble the sampler as shown in Figure 1. Check the COLIWASA to make sure it is functioning properly. Adjust the locking mechanism so that the neoprene stopper provides a thin closure.

2. If using a non-disposable COLIWASA, clean the COLIWASA according to procedures specified in SOP No. 002, General Equipment Decontamination. Place all sampling equipment on a plastic sheet next to the container to be sampled. Sample containers should be selected in accordance with the field sampling plan or Quality Assurance Project Plan.

3. Affix a completed sample container label to the appropriate sample container.

4. Wear appropriate PPE. Use a PID or FID to monitor airborne organic vapors and gases in the breathing zone. In most cases a PID is preferred because it is intrinsically safe, although an FID may be appropriate in some cases.

5. Record in the field logbook all exterior container markings, special conditions, and the type of opening through which the sample will be collected.

6. Using a permanent marker, make a unique identifying number on the container.

7. Locate an existing opening or a bung hole in the container, if possible.

8. Using non-sparking tools, a bung remover, or a remote drum opener, carefully remove the cover or bung from the container.

9. Place the COLIWASA in the open (sampling) position. The stopper rod handle should be in the T position and the rod should be pushed down until the handle rests against the locking block.

10. Slowly lower the COLIWASA into the liquid or slurry at a rate that permits the levels of the liquid or slurry inside and outside the COLIWASA tube to be about the same. If the liquid or slurry level in the COLIWASA tube is lower than that outside the COLIWASA tube, the sampling rate is too fast and will produce a nonrepresentative sample.

11. When the stopper reaches the bottom of the container, push the COLIWASA tube downward against the stopper to close it. Lock the COLIWASA tube in the closed position by turning the stopper rod handle from the T position until it is upright and one end rests tightly against the locking block.

12. Remove the COLIWASA tube from the container and wipe it with a disposable cloth.

13. Slowly discharge the sample into the labeled sample container. To do this, slowly pull the lower end of the stopper rod handle away from the locking block while the lower end of the COLIWASA tube is positioned in the sample container.

14. Ensure that a Teflon® liner is present in the sample container cap. Secure the cap tightly on the sample container. All containerized liquid and slurry samples should be evaluated in accordance with the "Sample Classification" section of SOP No. 019 (Packaging and Shipping Samples) to determine if they are hazardous samples; hazardous samples should be packaged and shipped in accordance with Dangerous Goods Regulations.

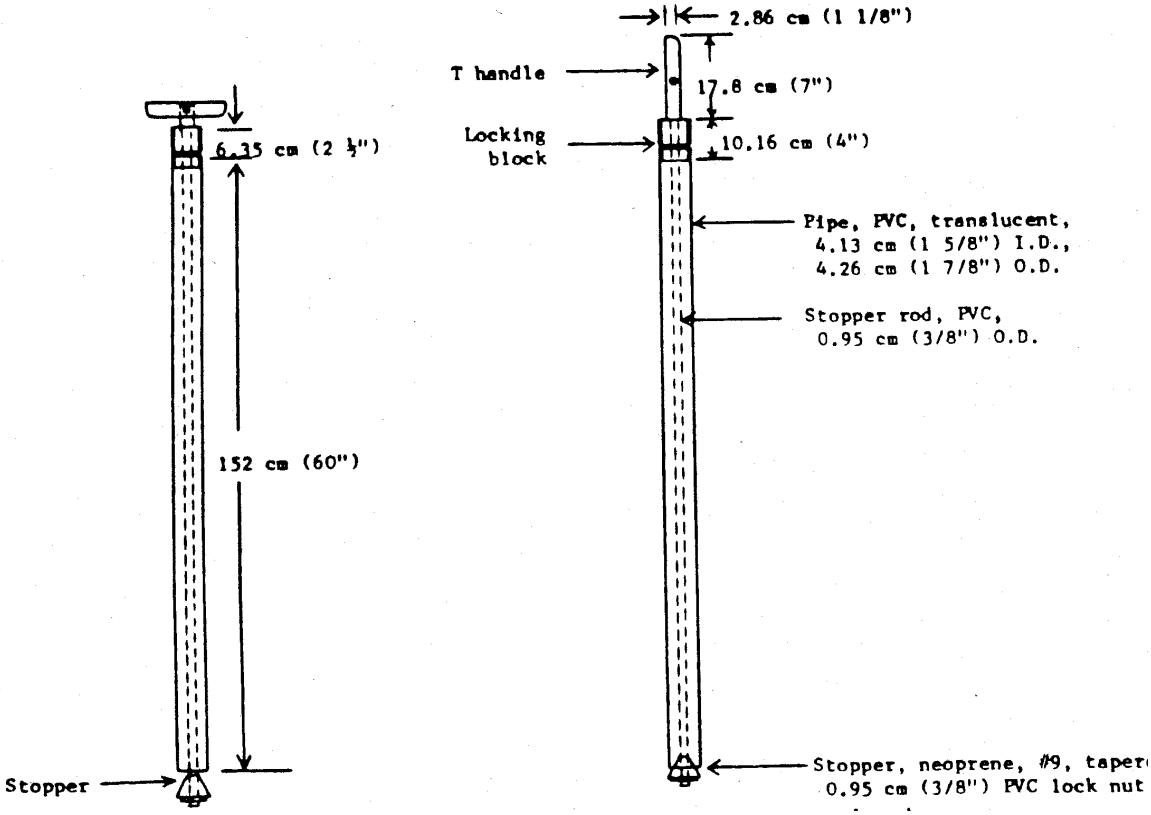
15. Replace the bung in the container or seal the opening in the container with plastic.

16. Complete all chain-of-custody forms and record sampling activities in the field logbook. Unless the sample is to be analyzed at the site, complete all sample packaging requirements in accordance with SOP No. 019, Packaging and Shipping Samples.

17. If a disposable COLIWASA was used, dispose of the device in an appropriate manner. Otherwise, unscrew the stopper rod handle of the COLIWASA tube and disengage the locking block. Decontaminate the COLIWASA tube on site or store the contaminated parts in a plastic storage tube for subsequent decontamination using the procedures in SOP No. 002, General Equipment Decontamination.

**FIGURE 1**

**COLIWASA**



**SOP APPROVAL FORM**

TETRA TECH, INC.

EMI OPERATING UNIT

ENVIRONMENTAL STANDARD OPERATING PROCEDURE

# PACKAGING AND SHIPPING SAMPLES

**SOP NO. 019**

**REVISION NO. 8**

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Quality Assurance Approved

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Date

# 1.0 BACKGROUND

In any sampling program, the integrity of a sample must be ensured from its point of collection to its final disposition. This standard operating procedure (SOP) describes procedures for packaging and shipping samples. Steps in the procedures should be followed to ensure sample integrity and to protect the welfare of persons involved in shipping and receiving samples.

## 1.1 PURPOSE

This SOP establishes the requirements and procedures for packaging and shipping nonhazardous environmental samples. It has been prepared in accordance with the U.S. Environmental Protection Agency (EPA) "Contract Laboratory Program Guidance for Field Samplers." Procedures described in this SOP should be followed for all routine sample packaging and shipping of nonhazardous samples. If procedures are to be modified for particular contract- or laboratory-specific requirements, modified procedures should be clearly described in site-specific plans such as work plans, field sampling plans (FSP), or quality assurance project plans (QAPP). Deviations from the procedures in this SOP must be documented in a field logbook. This SOP assumes that samples are already in the appropriate sample jars and that the sample jars are labeled.

***This SOP does not cover the packaging and shipment of Dangerous Goods or Hazardous Materials.*** The shipment of Dangerous Goods (by air) and Hazardous Materials (by ground) requires specialized training. If you have NOT received this training in the last 2 years, you are NOT qualified to package or ship these materials and may be personally liable for any damages or fines. Contact one of Tetra Tech's shipping experts for assistance. Instructions to access the training course, shipping experts, and health and safety (H&S) contacts, and general information on packaging and shipping hazardous substances and dangerous goods can be obtained by checking the links provided in Section 1.4 (References) and communicating with appropriate Tetra Tech H&S contacts listed on the EMI Operating unit internal H&S web site.

## 1.2 SCOPE

This SOP applies to packaging and shipping of environmental and nonhazardous samples. This SOP does not address shipping dangerous goods or hazardous materials.

## 1.3 DEFINITIONS

**Airbill:** An airbill is a shipping form (such as a FedEx shipping form) acquired from the commercial shipper and is used to document shipment of the samples from the sampler to the designated analytical laboratory (see Figure 1).

**Blank:** A blank is any sample that is used to assess cross-contamination from sampling and sample management procedures. A typical blank sample will consist of distilled or deionized (DI) water (water sampling) or an air filter cartridge (air sampling) that is then analyzed by the laboratory to evaluate whether cross-contamination has been introduced. Each blank is assigned its own unique sample number. Blanks collected in the field include trip blanks, field blanks, and equipment blanks, all intended to assess potential cross-contamination. For example, a trip blank checks for contamination during sample handling, storage, and shipment from the field to the laboratory. Field blanks assess the contamination of water or soil from ambient air. Equipment blanks (also known as rinse blanks) assess contamination from incomplete decontamination procedures.

**Chain-of-Custody form:** A chain-of-custody form is used to document the transfer of custody of samples from the field to the designated analytical laboratory (see Figure 2). The chain-of-custody form is critical to the chain-of-custody process and is used to identify the samples in each shipping container to be shipped or delivered to the laboratory for chemical or physical (geotechnical) analysis. A copy of the chain-of-custody form is shipped with the samples and accompanies them from sampler to laboratory (see Figure 3).

**Custody seal:** A custody seal is a tape-like seal and is used to indicate that samples are intact and have not been disturbed during shipping or transport after the samples have been released from the sampler to the shipper (see Figure 4). The custody seal is part of the chain-of-custody process and is used to prevent tampering with samples after they have been packaged for shipping (see Figure 5).

**Environmental samples:** Environmental samples include drinking water, groundwater, surface water, soil, sediment, treated municipal and industrial wastewater effluent, indoor and ambient air, nonhazardous bulk materials, soil gas, dust, asbestos, and biological specimens. Environmental samples typically contain low concentrations of contaminants and, when handled, require only limited precautionary procedures.

**Nonhazardous samples:** Nonhazardous samples are those samples that do not meet the definition of a hazardous sample AND do not need to be packaged and shipped in accordance with the International Air Travel Association's (IATA) "Dangerous Goods Regulations" (DGR) or U.S. Department of Transportation's "Hazardous Materials Regulations" defined in Title 49 *Code of Federal Regulations* (CFR).

The following definitions are provided to further distinguish environmental and nonhazardous samples from dangerous goods and hazardous samples:

**Dangerous goods:** Dangerous goods are articles or substances that can pose a significant risk to health, safety, or property when transported by air; they are classified as defined in Section 3 of the DGR (IATA 2020).

**Hazardous samples:** Hazardous samples include dangerous goods and hazardous substances. Hazardous samples shipped by air should be packaged and labeled in accordance with procedures specified by the DGR; ground shipments should be packaged and labeled in accordance with the Hazardous Material Regulations.

**Hazardous substance:** A hazardous substance is any material, including its mixtures and solutions, that is listed in 49 CFR 172.101 and its quantity, in one package, equals or exceeds the reportable quantity listed in Table 1 to Appendix A of 49 CFR 172.101.

## 1.4 REFERENCES

General Awareness, H&S Contacts, and Course Training Information (Tetra Tech, Inc., EMI Operating Unit. Intranet) On-line address: https://int.tetratech.com/sites/EMI/hs/Pages/Dangerous-Goods-Shipping.aspx

International Air Transport Association (IATA). 2020. "Dangerous Goods Regulations. 2020." For sale at: https://www.iata.org/en/publications/dgr/. Updated annually, with new edition available late in year.

U.S. Environmental Protection Agency (EPA). 40 CFR, 763 Subpart F, Asbestos Hazards Emergency Response Act (AHERA).

EPA. 2014. "Contract Laboratory Program Guidance for Field Samplers." EPA 540-R-014-013. October. On-line address: https://www.epa.gov/sites/production/files/2015-03/documents/samplers_guide.pdf.

EPA. 2020. "Packing, Marking, Labeling and Shipping of Environmental and Waste Samples." EPA Region 4, LSASDPROC-209-R4. February 23. On-line address: https://www.epa.gov/sites/production/files/2015-06/documents/Shipping-Environmental-and-Waste-Samples.pdf

## 1.5 REQUIREMENTS AND RESOURCES

The procedures for packaging and shipping samples require the following:

- Coolers (insulated ice chest) or other shipping containers appropriate to sample type
- Ice
- Bubble wrap or similar cushioning material
- Chain-of-custody forms and seals
- Airbills

- Resealable plastic bags for sample jars and ice
- Tape (strapping and clear)
- Large plastic garbage bags for lining the cooler
- Temperature blank sample bottle filled with distilled water can be included in the cooler if appropriate to sample type
- Trip blank samples used to check for volatile contamination during sample handling in the field should accompany sample containers during shipment from laboratory to field (empty containers) and from field to laboratory (filled containers). It should remain in the cooler with sample containers during the sampling event. Trip blanks should be requested from the laboratory when containers are initially ordered.

## 2.0 PROCEDURES

The following procedures apply to packaging and shipping nonhazardous and environmental samples.

### 2.1 PACKAGING SAMPLES

After they have been appropriately containerized and labeled, environmental samples should be packaged as described in this section. This section covers procedures for packing samples for delivery by commercial carrier (air or ground) and hand delivery of environmental samples (by employee or courier), as well as shipping asbestos and air quality samples. Note that these instructions are general; samplers also should be aware of client-specific requirements concerning the placement of custody seals or other packaging provisions.

#### 2.1.1 Packaging Samples for Delivery by Commercial Carrier (Air or Ground)

Samples shipped by commercial carriers should be packed for shipment using the following procedures and in compliance with all carrier requirements:

**Preparing the sample:**

1. Allow a small amount of headspace in all bottles, or as instructed by the laboratory (except volatile organic compound [VOC] containers with a septum seal) to compensate for any changes in pressure and temperature during transfer.
2. Be sure the lids on all bottles are tight (will not leak). Lids maybe taped or sealed with custody seals as added protection or as required. For any sample containers that are not marked with a tare weight by the laboratory, cover the completed sample label on the container with clear tape to protect the label.
3. Place sample containers in resealable plastic bags.

**Preparing the cooler:**

1. Secure and tape the drain plug of the cooler with fiber or duct tape.
2. Line the cooler with a large plastic garbage bag before samples, ice, and absorbent packing material are placed in the cooler.
3. Wrap the sample containers in bubble wrap or line the cooler (bottom and sides) with a cushioning material to prevent breakage of bottles or jars during shipment.
4. If required by the laboratory for the analytical method, add a sufficient quantity of ice to the cooler to cool samples to 4 °C (± 2 °C). Ice should be double bagged in resealable plastic bags to prevent the melted ice from leaking out. If required, include one temperature blank (a sample bottle filled with distilled water) per cooler.

5. For VOC samples only, include one trip blank for VOC analysis per shipment matrix in each cooler.

6. Fill all remaining space between the bottles or jars with bubble wrap.

7. As each container is placed in the cooler, verify the sample information on the chain-of-custody form. The samples listed on the chain-of-custody form must match exactly with the contents of the cooler.

8. Securely fasten the top of the large garbage bag with tape (preferably plastic electrical tape).

9. If more than one cooler is being shipped, mark each cooler as “1 of 2,” “2 of 2,” and so forth.

10. Place the chain-of-custody forms (see Figure 2) into a resealable plastic bag, and tape the bag to the inner side of the cooler lid (see Figure 3). If you are shipping more than one cooler, copy the chain-of-custody form so that there is one copy of all forms in each cooler. The samples listed on the chain-of-custody form must match exactly with the contents of the cooler. Tape any instructions for returning the cooler to the inside of the lid.

11. Close the lid of the cooler and tape it shut by wrapping strapping tape around both ends and hinges of the cooler at least once.

12. Place two signed custody seals (see Figure 4) on opposite sides of the cooler, ensuring that each one covers the cooler lid and side of the cooler (see Figure 5; note that in contrast to the figure, the seals should be placed on the opposite sides of the cooler and offset from each other, rather than directly across from each other as shown in Figure 5). Place clear plastic tape over the custody seals so that the cooler cannot be opened without breaking the seal.

13. Shipping containers should be marked "THIS END UP.” Arrow labels, which indicate the proper upward position of the container, may also be affixed to the container. As appropriate, the containers should also be labeled for Saturday delivery or other special requirements.

14. Ship samples overnight using a commercial carrier such as FedEx. As a best practice, electronic sample shipping labels should be prepared by the shipping agency’s employees, at the direction of Tetra Tech employees or sampling personnel. This allows the sampling personnel to confirm special shipping requirements, such as Saturday delivery, and verify that samples will be shipped that day (that is, the last shipment of the day has not already occurred). If this is not possible, the airbill can be prepared by hand (see Figure 1), but samples should still be handed over directly to shipping agency employees and shipping details should be verified. The shipping label should be placed on the outside of the container.

15. A copy of the receipt with sample tracking number should be retained by the sampling personnel and delivery should be verified the next day.

### 2.1.2 Hand Delivery of Environmental Samples (by Employee or Courier)

Samples hand-delivered to the laboratory should be packed for shipment using the following procedures:

**Preparing the sample:**

1. Bottles can be filled completely with sample (required for VOC containers with a septum seal).

2. Be sure the lids on all bottles are tight (will not leak).

**Preparing the cooler:**

1. Secure and tape the drain plug of the cooler with fiber or duct tape.

2. Wrap the sample containers in bubble wrap or line the cooler (bottom and sides) with a cushioning material to prevent breakage of bottles or jars during shipment.

3. As each container is placed in the cooler, verify the sample information on the chain-of-custody form. The samples listed on the chain-of-custody form must match exactly with the contents of the cooler.

4. If required for by the laboratory for the analytical method, add a sufficient quantity of ice to the cooler to cool samples to 4 °C. Ice should be double bagged in resealable plastic bags to prevent the melted ice from leaking out. If required, include one temperature blank (a sample bottle filled with distilled water) per cooler.

5. For VOC samples only, include one trip blank for VOC analysis per shipment matrix in each cooler.

6. If more than one cooler is being shipped, mark each cooler as "1 of 2," "2 of 2," and so forth.

7. Place the chain-of-custody form (see Figure 2) in a resealable plastic bag and tape to the inside of the cooler lid (see Figure 3), close the lid, and seal with custody seals (see Figure 5; note that in contrast to the figure, the seals should be placed on the opposite sides of the cooler and offset from each other, rather than directly across from each other as shown in Figure 5). Place clear plastic tape over the custody seals so that the cooler cannot be opened without breaking the seal. Transfer the cooler to the courier. When samples will be delivered directly to the laboratory, it is sufficient to close the cooler and hand-deliver it with the chain-of-custody form.

8. Include any instructions for returning the cooler to the inside of the lid.

9. If the cooler is being transferred to a courier, the shipping containers should be marked "THIS END UP," and arrow labels, which indicate the proper upward position of the container should be affixed to the container.

### 2.1.3 Shipping Asbestos Samples

Asbestos samples shipped by commercial carriers should be packed for shipment using the following procedures and in compliance with all carrier requirements:

1. Place each asbestos sample in a small resealable plastic bag or Whirl-pak sealable bag. Seal the bags carefully and place the sample bags in a larger resealable plastic bag.

2. Select a rigid shipping container and pack the samples upright in a noncontaminating, nonfibrous medium such as a bubble pack to minimize excessive movement during shipping.

3. Avoid using expanded polystyrene because of its static charge potential. Also avoid using particle-based packaging materials because of possible contamination.

4. Affix custody seals to the samples or outer sample bag so that the bags cannot be opened without breaking the seal.

5. Insert the chain-of-custody form in the box. Include a shipping bill and a detailed listing of samples shipped, their descriptions and all identifying numbers or marks, sampling data, shipper's name, and contact information.

6. Ship bulk samples in a separate container from air samples. Bulk samples and air samples delivered to the analytical laboratory in the same container will be rejected.

7. For each sample set, designate which are the ambient samples, which are the abatement area samples, which are the field blanks, and which is the sealed blank if sequential analysis is to be performed.

8. Hand-carry samples to the laboratory in an upright position if possible; otherwise, choose that mode of transportation least likely to shake the samples in transit.

9. Address the package to the laboratory sample coordinator by name when known and alert him or her of the package description, shipment mode, and anticipated arrival as part of the chain-of-custody and sample tracking procedures. This information will also help the laboratory schedule timely analysis for the samples when they are received.

### 2.1.4 Shipping Air Samples

Packaging and shipping requirements for air samples vary depending on the media used to collect the samples and the analyses required. Sampling media typically include Summa canisters and Tedlar bags for whole air samples, filters for metals and particulate matter, and sorbent tubes for organic contaminants. This section of the SOP provides general guidelines for packaging and shipping air samples collected using these media. The project FSP or QAPP should also be reviewed for any additional project-specific requirements or instructions.

**Summa Canister Samples**

1. Close the canister valve by tightening the knob clockwise or flipping the toggle switch. Replace the brass cap on the canister inlet.

2. If a flow controller was used to collect the air sample over a specified time interval, the flow controller should be removed before replacing the brass cap.

3. Fill out the sample tag on the canister with the sample number and the date and time of collection. Include the identification number of the flow controller on the sample tag if one was used. Make sure the information on the sample tag matches the chain-of-custody form.

4. Complete the chain-of-custody form. In addition to the information normally included, the form should include the following data: sample start and stop dates and times; initial and final Summa canister vacuum readings; Summa canister identification number; and flow controller identification number.

5. Package the Summa canister (and flow controller) in its original shipping box with the original packaging material. Tape the box shut and apply custody seals if required. Note: Summa canisters should never be packaged with ice.

6. Summa canister shipments typically include several canisters, and may include more than one shipping box. The chain-of-custody form for the shipment should be sealed within one of the shipping boxes. If more than one box is being shipped, mark each box as "1 of 2," "2 of 2," and so forth.

7. Ship the samples by a method that will meet the holding time. Summa canister samples should be analyzed within 30 days of sample collection.

**Tedlar Bag Samples**

1. Before removing it from the sample port, close the Tedlar bag by tightening the valve clockwise. The bag should only be approximately half-full to allow for pressure changes during shipping and handling of the sample. Keep the Tedlar bag out of direct sunlight to preserve the sample.

2. Fill out the label on the bag with the sample number and the date and time of sample collection. Make sure the information on the label matches the chain-of-custody form.

3. Complete the chain-of-custody form.

4. Package the Tedlar bag in a shipping box with appropriate packing material to prevent the bag from being punctured or damaged. Multiple bags can be packaged in the same box. Tape the box shut and apply custody seals if required. Note: Tedlar bag samples should not be cooled or packaged with ice, although they can be shipped in an ice chest to protect the samples.

5. Tedlar bag shipments may include more than one shipping box. The chain-of-custody form for the shipment should be sealed within one of the shipping boxes. If more than one box is being shipped, mark each box as "1 of 2," "2 of 2," and so forth.

6. Ship the samples using priority overnight delivery. Tedlar bag samples should be analyzed within 3 days of sample collection.

**Filter Cassette Samples**

1. Disconnect the filter cassette from the air sampling pump and replace the plastic caps on the inlet and outlet openings.

2. Attach a label to the sample that includes the sample number and the date and time of sample collection. Make sure the information on the label matches the chain-of-custody form.

3. Complete the chain-of-custody form. In addition to the information normally included, the form should include the following data: sample start and stop dates and times; initial and final air flow rates (or average flow rate); volume of air sampled; and sampling pump identification number.

4. Package the filter cassettes in a shipping box (such as a FedEx box). Use an appropriate packing material (such as bubble wrap) to separate the samples and prevent damage.

5. Place the chain-of-custody form within the box, seal the box, and apply custody seals if required. Filter cassette samples typically do not need to be cooled, but check the field sampling plan (FSP) or Quality Assurance Project Plan (QAPP) for project-specific requirements.

6. Ship the samples by a method that will meet the holding time.

**Sorbent Tube Samples**

1. Disconnect the sample tube from the air sampling pump and seal both ends of the tube with plastic caps.

2. Complete a sample label that includes the sample number and the date and time of sample collection. Make sure the information on the label matches the chain-of-custody form.

3. If the tube is small and the label cannot be attached to the tube, the tube can be placed in a small resealable plastic bag and the label can be attached to the bag or placed inside the bag with the tube.

4. Complete the chain-of-custody form. In addition to the information normally included, the form should include the following data: sample start and stop dates and times; initial and final air flow rates (or average flow rate); volume of air sampled; and sampling pump identification number.

5. Packaging requirements for the sample tubes will depend on the analysis required, and the sampler should check the FSP or QAPP for project-specific requirements (for example, tubes may need to be wrapped in aluminum foil to prevent exposure to light). Packaging containers and methods include (1) shipping boxes (as described under filter cassette samples), (2) small sample coolers filled with double-bagged ice, and (3) small sample coolers filled with blue (reusable) ice.

6. Place the chain-of-custody form within the box or container, seal the box or container, and apply a custody seal if required.

7. If coolers are used for shipping, tape instructions for returning the cooler to the inside of the lid.

8. Ship the samples by a method that will meet the holding time.

**Polyurethane Foam (PUF) Tube Samples**

1. Disconnect the PUF tube from the air sampling pump and wrap the tube in aluminum foil.

2. Attach a label to the wrapped sample tube that includes the sample number and the date and time of sample collection. Make sure the information on the label matches the chain-of-custody form.

3. Wrap the PUF tube in bubble wrap and place the tube in a glass shipping jar.

4. Complete the chain-of-custody form. In addition to the information normally included, the form should include the following data: sample start and stop dates and times; initial and final air flow rates (or average flow rate); volume of air sampled; and sampling pump identification number.

5. Package the PUF tube jars in a cooler that is filled with double-bagged ice. Use bubble wrap or other cushioning material to separate the samples and prevent breakage.

6. Place the chain-of-custody form within the cooler, seal the cooler, and apply a custody seal if required.

7. If coolers are used for shipping, tape instructions for returning the cooler to the inside of the lid.

8. Ship the samples by a method that will meet the holding time. Samples collected in PUF tubes typically must be extracted within 7 days of collection.

## 2.2 SHIPPING DOCUMENTATION FOR SAMPLES

Airbills, chain-of-custody forms, and custody seals must be completed for each shipment of nonhazardous environmental samples.

Field staff collecting samples should also review their field work plans to confirm what documentation must be completed during each sampling event, including client-specific requirements. For example, some EPA programs have a specific requirement to use Scribe software, an environmental data management system, to create sample documentation, electronically input information into Traffic Report or chain-of-custody forms, and enter other data.

- The Scribe software can be accessed from the EPA Environmental Response Team (ERT) at the following address: http://www.ertsupport.org/scribe_home.htm
- The ERT User Manual for Scribe, reference, and training materials can be accessed from the Scribe Support Web site at the following address: http://www.epaosc.org/scribe

Note that some laboratories must routinely return sample shipping coolers within 14 calendar days after the shipment has been received. Therefore, the sampler should also include instructions for returning the cooler with each shipment, when possible. The sampler (not the laboratory) is responsible for paying for return of the cooler and should include shipping airbills bearing the sampler's shipping account number, as well as a return address to allow for return of the cooler. Samplers should use the least expensive option possible for returning coolers.

## 2.3 SHIPMENT DELIVERY AND NOTIFICATION

A member of the field sampling team must contact the laboratory to confirm it accepts deliveries on any given day, especially Saturdays. In addition, samplers should ensure the laboratory has been notified in advance of the pending shipment and notify any additional parties as required. The sampler needs to know the laboratory's contact name, address, and telephone number and be aware of the laboratory's requirements for receiving samples.

In addition, samplers should be aware of the sample holding times, shipping company's hours of operation, shipping schedule, and pick-up and drop-off requirements to avoid delays in analytical testing.

### Priority Overnight Delivery

Priority overnight delivery is typically the best method for shipment. Delays caused by longer shipment times may cause the sample temperature to rise above the acceptable range of 4° C (± 2 ° C) and technical holding time may expire, which in turn may compromise sample integrity and require recollection of

samples. If sample delivery procedures are to be modified for particular contract- or laboratory-specific requirements, the procedures should be clearly described in site-specific plans such as work plans, FSPs, or QAPPs.

**Saturday Delivery**

If planning to ship samples for Saturday delivery, the laboratory must be contacted in advance to confirm it will accept deliveries on Saturdays or arrange for them to be accepted. In addition, samplers should ensure the laboratory has been notified in advance of the pending shipment and notify any additional parties as required.

## 2.4 HEALTH AND SAFETY CONSIDERATIONS

In addition to the procedures outlined in this SOP, all field staff must be aware of and follow the health and safety practices that result from the Activity Hazard Analyses (AHA) for the project. The AHAs include critical safety procedures, required controls, and minimum personal protective equipment necessary to address potential hazards. The hazards specific to project tasks must be identified and controlled to the extent practicable and communicated to all project personnel via the approved, project-specific health and safety plan (HASP).

## 3.0 POTENTIAL PROBLEMS

The following potential problems may occur during sample shipment:

- Leaking package. If a package leaks (either from broken sample containers or melting ice), the carrier may open the package and return the package. Special care should be taken during sample packaging to minimize potential leaks.

- Improper labeling and marking of package. If mistakes are made in labeling and marking the package, the carrier will most likely notice the mistakes and return the package to the shipper, thus delaying sample shipment. A good practice is to have labels, forms, and container markings double checked by a member of the field team.

- Bulk samples and air samples delivered to the analytical laboratory in the same container. If samples are combined in this way, they will be rejected. Always ship bulk samples in separate containers from air samples.

- Issues in packing asbestos samples. When asbestos samples are shipped, avoid using expanded polystyrene because of its static charge potential. Also avoid using particle-based packaging materials with asbestos samples because of possible contamination.

- Improper, misspelled, or missing information on the shipper's declaration. The carrier will most likely notice these errors as well and return the package to the shipper. A good practice is to have another field team member double check this information.

- Missed drop off time or wrong location. Missing the drop off time or having the wrong location identified for drop off will delay delivery to the laboratory and may cause technical holding times to expire. Establish the time requirements in advance of completing the field effort and be sure and provide some contingency time for potential delays such as traffic or checking and redoing paperwork.

- Incorrectly packaging samples for analysis at multiple laboratories. For example, inorganic samples may be shipped to one laboratory for analysis, while organic samples may need to be shipped to another laboratory. All field staff should be aware which samples are to be shipped to which laboratory when they package samples for multiple types of analysis.

- Holidays or weather-related delays. Be aware of holidays and weather forecasts that could cause delays in delivery. Delays caused by longer shipping times may cause technical holding times to expire, which in turn may compromise sample integrity or require recollection of samples.

- Not noting field variances in field logbook. Field variances should be noted in the field logbook and the project manager notified. Common field variances include:

    - Less sample volume collected than planned. Notify appropriate staff and the laboratory to ensure there is an adequate amount for analysis.

    - Sample collected into incorrect jar because of broken or missing bottle-ware. Notify appropriate laboratory staff to ensure there is no confusion regarding the analysis of the sample.

**FIGURE 1**

**EXAMPLE OF A FEDEX US AIRBILL FOR LOW-LEVEL ENVIRONMENTAL SAMPLES**


**Filling Out the FedEx US Airbill**

- The sender *must complete* the following fields on the pre-printed airbill:
  - Section 1: Date
  - Section 1: Sender's FedEx Account Number (available from your office administrator)
  - Section 1: Sender's Name, Company, Address, and Phone Number
  - Section 2: Internal Billing Reference (Project Number) (this field may not be present on newer airbills)
  - Section 3: Recipient's Name, Company, Address, and Phone Number
  - Section 4: Express Package or Freight Services (Priority Overnight)
  - Section 5: Packaging (usually "Other," your own packaging)
  - Section 6: Special Handling (Saturday delivery if prearranged with receiving laboratory; "No" dangerous goods contained in shipment)
  - Section 7: Payment ("Bill to Sender")
  - Section 7: Total Number of Packages
  - Section 7: Total Weight (completed by FedEx employee)
  - Section 8: Delivery Signature Options ("No Signature Required")

**Completing a Sample Chain-of-Custody Form** (See Also Section 2.2 on SCRIBE for Forms)

After samples have been collected, they will be maintained under chain-of-custody procedures. These procedures are used to document the transfer of custody of the samples from the field to the designated

analytical laboratory. The same chain-of-custody procedures will be used for the transfer of samples from one laboratory to another, if required.

The field sampling personnel will complete a Chain-of-Custody and Request for Analysis (CC/RA) form for each separate container of samples to be shipped or delivered to the laboratory for chemical or physical (geotechnical) analysis. These forms are often triplicate, carbonless forms. Care should be taken when completing the form that all copies are legible—PRESS FIRMLY WHEN WRITING. Information on the form will include:

1. Project identification (ID) (for example, contract and task order number);
2. Project Contract Task Order (CTO) number;
3. Laboratory Project Order (PO) number;
4. Tetra Tech Technical Contact;
5. Tetra Tech Project Manager;
6. Laboratory name;
7. Field sampler names;
8. Field sampler signature;
9. Sample ID;
10. Date and time of sampling;
11. Sample matrix type;
12. Sample preservation method; note “NONE” if no preservatives;
13. Number and types of containers per sample;
14. Sample hazards (if any);
15. Requested analysis;
16. Requested sample turnaround time or any special remarks (for example, possible presence of free product or high screening concentrations);
17. Page __ of __;
18. Method of shipment;
19. Carrier/waybill number (if any);
20. Signature, name, and company of the person relinquishing the samples and the person receiving the samples when custody is transferred;

21. Date and time of sample custody transfer;

22. Condition of samples when they are received by the laboratory.

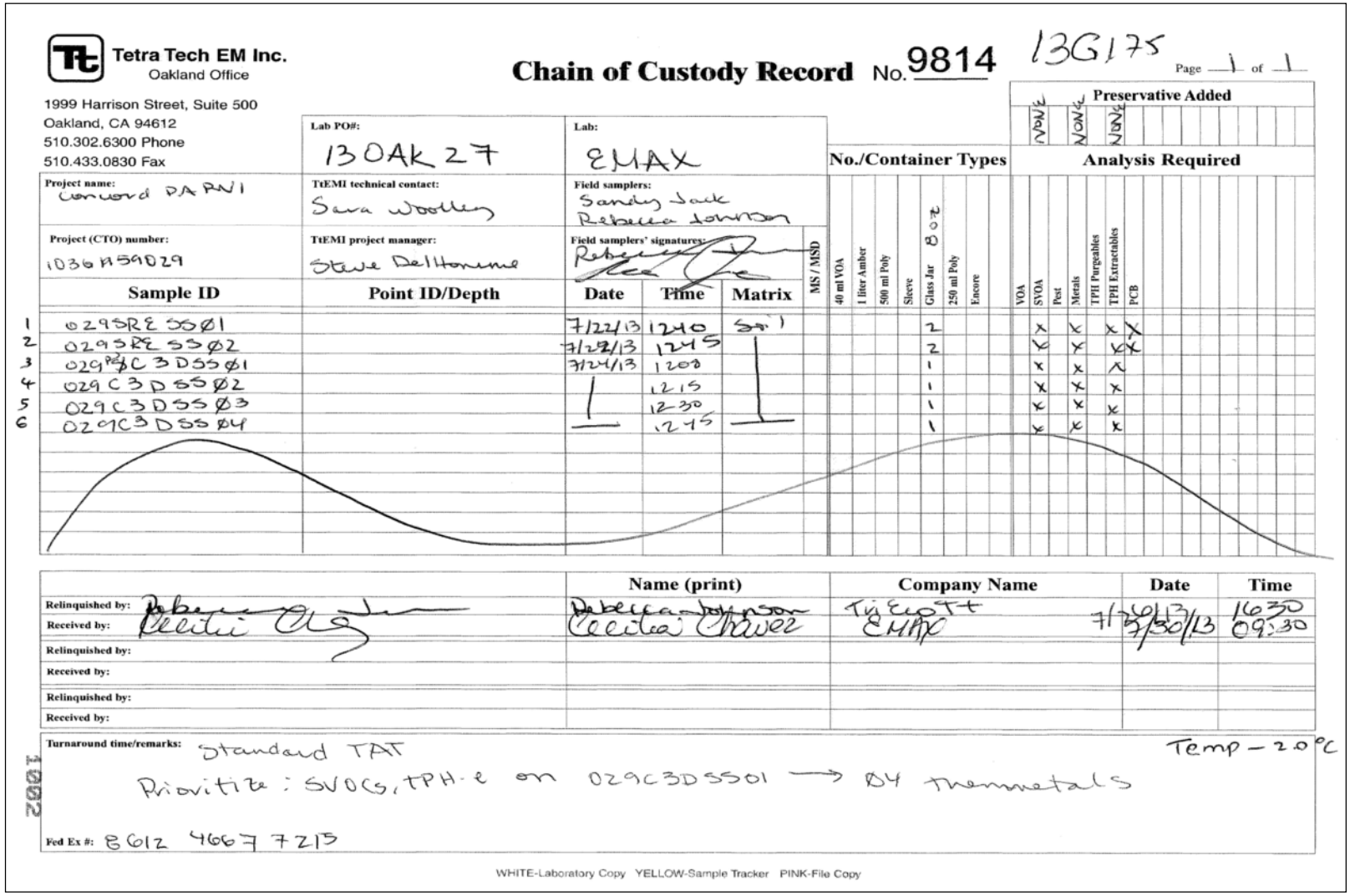
The sample collector will cross out any blank space on the CC/RA form below the last sample number listed on the part of the form where samples are listed.

The sampling personnel whose signature appears on the CC/RA form is responsible for the custody of a sample from time the sample is collected until the custody of the sample is transferred to a designated laboratory, a courier, or to another Tetra Tech employee for transporting a sample to the designated laboratory. A sample is considered to be in custody when the custodian: (1) has direct possession of it; (2) has plain view of it; or (3) has securely locked it in a restricted access area.

Custody is transferred when both parties to the transfer complete the portion of the CC/RA form under "Relinquished by" and "Received by" or a sample is left at a FedEx facility pending shipment. Signatures, printed names, company names, and date and time of custody transfer are required. When custody is transferred, the Tetra Tech sampling personnel who relinquished the samples will retain the third sheet (pink copy) of the CC/RA form. When the samples are shipped by a common carrier, a Bill of Lading supplied by the carrier will be used to document the sample custody, and its identification number will be entered on the CC/RA form. Receipts of Bills of Lading will be retained as part of the permanent documentation in the Tetra Tech project file.

**FIGURE 2**

**EXAMPLE OF A CHAIN-OF-CUSTODY FORM (WHITE COPY)**



Tetra Tech EM Inc.
Oakland Office
1999 Harrison Street, Suite 500
Oakland, CA 94612
510.302.6300 Phone
510.433.0830 Fax

**Chain of Custody Record** No. 9814 13G175 Page 1 of 1

Lab PO#: 130Ak27 | Lab: EMAX

Project name: Concord PA PN1 | TtEMI technical contact: Sara Woolley | Field samplers: Sandy Jack, Rebecca Johnson

Project (CTO) number: 103G159029 | TtEMI project manager: Steve DeHoratius | Field samplers' signatures:

| | Sample ID | Point ID/Depth | Date | Time | Matrix | MS / MSD | No./Container Types: 40 ml VOA | 1 liter Amber | 500 ml Poly | Sleeve | Glass Jar 8 oz | 250 ml Poly | Encore | Preservative Added: NONE | NONE | NONE | Analysis Required: VOA | SVOA | Pest | Metals | TPH Purgeables | TPH Extractables | PCB |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 1 | 029SRE SS01 | | 7/22/13 | 1240 | Soil | | | | | | 2 | | | | | | X | X | X | | X | X | |
| 2 | 029SRE SS02 | | 7/22/13 | 1245 | | | | | | | 2 | | | | | | X | X | X | | X | X | |
| 3 | 029PBC 3D SS01 | | 7/24/13 | 1200 | | | | | | | 1 | | | | | | X | X | X | | X | | |
| 4 | 029 C 3D SS02 | | | 1215 | | | | | | | 1 | | | | | | X | X | X | | X | | |
| 5 | 029C3D SS03 | | | 1230 | | | | | | | 1 | | | | | | X | X | X | | X | | |
| 6 | 029C3D SS04 | | | 1245 | | | | | | | 1 | | | | | | X | X | X | | X | | |

| | Name (print) | Company Name | Date | Time |
|---|---|---|---|---|
| Relinquished by: | Rebecca Johnson | TtEMI | 7/26/13 | 1630 |
| Received by: | Cecilia Chavez | EMAX | 7/30/13 | 09:30 |
| Relinquished by: | | | | |
| Received by: | | | | |
| Relinquished by: | | | | |
| Received by: | | | | |

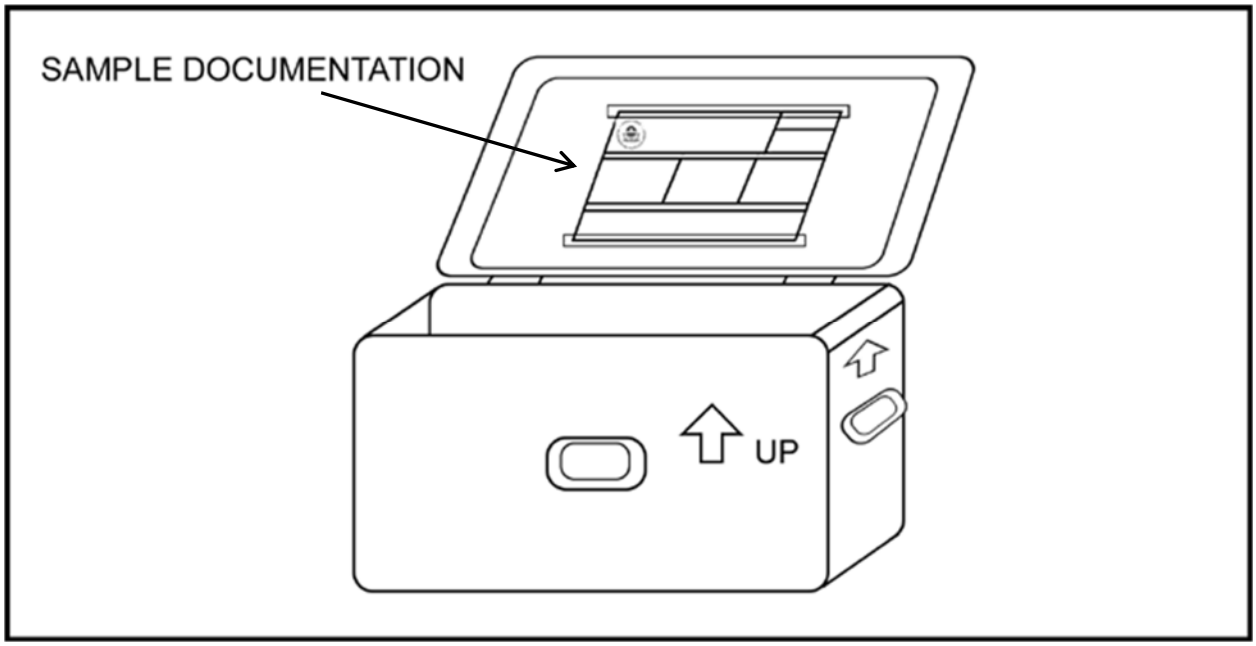
Turnaround time/remarks: Standard TAT Temp – 20°C
Prioritize: SVOCs, TPH-e on 029C3DSS01 → 04 thenmetals

Fed Ex #: 8612 4667 7215

WHITE-Laboratory Copy YELLOW-Sample Tracker PINK-File Copy

**FIGURE 3**

**EXAMPLE OF A SAMPLE COOLER WITH ATTACHED DOCUMENTATION**




Source: U.S. Environmental Protection Agency. 2014.

Place the necessary paperwork (chain-of-custody form, cooler return instructions, and associated paperwork) in the shipping cooler or acceptable container. All paperwork must be placed in a plastic bag or pouch and then secured to the underside of the shipping container lid.

**FIGURE 4**

**EXAMPLE OF A CUSTODY SEAL**

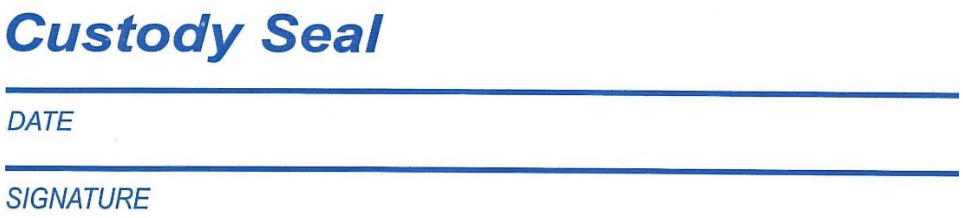
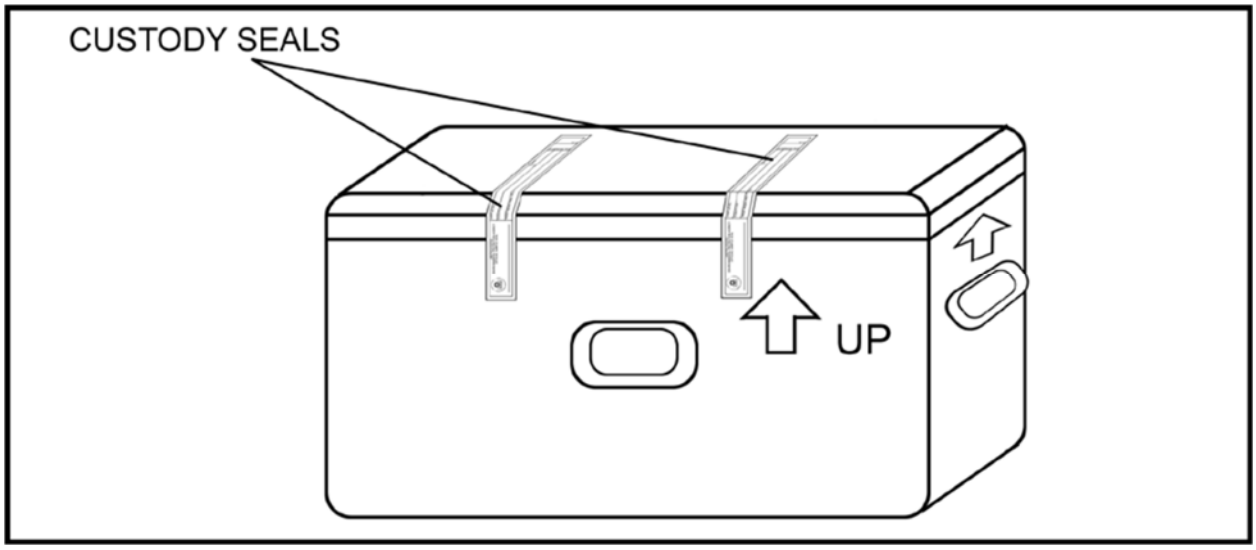


**FIGURE 5**

**EXAMPLE OF SHIPPING COOLER WITH CUSTODY SEALS**




Source: U.S. Environmental Protection Agency. 2014.

Please note that the two seals typically are affixed to opposite sides of the cooler and offset from each other, although the offset is not depicted on the EPA figure above.

**SOP APPROVAL FORM**

TETRA TECH, INC.

EMI OPERATING UNIT

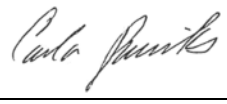
ENVIRONMENTAL STANDARD OPERATING PROCEDURE

# RECORDING NOTES IN FIELD LOGBOOKS

**SOP NO. 024**

**REVISION NO. 3**

Last Reviewed: July 2020



Quality Assurance Approved

*July 2, 2020*

Date

# 1.0 BACKGROUND

Complete and accurate field documentation is critical to a successful project and the field logbook is an important tool to support field documentation needs. The field logbook should include detailed records of all field activities, document interviews with people, and record observations of conditions at a site. Entries should be described in a level of detail to allow personnel to reconstruct, after the fact, activities and events that occurred during their field assignments. Furthermore, entries should be limited to facts. Avoid speculation related to field events and do not record hearsay or unfounded information that may be presented by other parties during field activities. For example, do not record theories regarding the presence or absence of contamination when you are collecting field screening data or speculation regarding the reasons for a property owner's refusal to grant access for sampling.

Field logbooks are considered accountable documents in enforcement proceedings and may be subject to review. Therefore, the entries in the logbook must be accurate and detailed, but should not contain speculative information that could conflict with information presented in subsequent project deliverables and correspondence. Also be aware that the field logbooks for a site may be a primary source of information for depositions and other legal proceedings that may occur months or years after field work is complete and long after our memories have faded. The accuracy, neatness, and completeness of field logbooks are essential for recreating a meaningful account of events.

Field notes may also be recorded digitally, using a variety of software programs. The requirements and use of digital recording programs is not addressed in this standard operating procedure (SOP) because many items are unique to the selected software system. However, many of the principles discussed in this SOP will apply to the digital recording of field notes.

## 1.1 PURPOSE

The purpose of this SOP is to provide guidance to ensure that field logbook documentation collected during field activities meets all requirements for its later use. Among other things, field logbooks may be used for:

- Identifying, locating, labeling, and tracking samples
- Recording site activities and the whereabouts of field personnel throughout the day
- Documenting any deviations from the project approach, work plans, quality assurance project plans, health and safety plans, sampling plans, and any changes in project personnel
- Recording arrival and departure times for field personnel each morning and evening and weather conditions each day

- Describing photographs taken during the project.

In addition, the data recorded in the field logbook may later assist in the interpretation of analytical results. A complete and accurate logbook also aids in maintaining quality control, because it can verify adherence to project scope and requirements.

## 1.2 SCOPE

This SOP establishes the general requirements and procedures for documenting site activities in the field logbook.

## 1.3 DEFINITIONS

None.

## 1.4 REFERENCES

Compton, R.R. 1985. *Geology in the Field*. John Wiley and Sons. New York, NY.

## 1.5 REQUIREMENTS AND RESOURCES

The following items are required for field notation:

- Bound (sewn) notebooks
- Ballpoint pens or Sharpies with permanent waterproof ink
- 6-inch ruler (optional)

Field logbooks should be bound (sewn) with water-resistant and acid-proof covers, and each page should have preprinted lines or grids and numbered pages. They should be approximately $7^1/_2$ by $4^1/_2$ inches or $8^1/_2$ by 11 inches in size. Loose-leaf sheets are not acceptable for use as a field logbook, although logs and field forms used to record field measurements and data are acceptable as loose-leaf sheets maintained in a three-ring binder with numbered pages, as a supplement to the logbook. If notes are written on loose paper, they must be transcribed as soon as possible into a bound field logbook by the same person who recorded the notes originally.

Ideally, distribution of logbooks should be controlled by a designated person in each office. This person assigns a document control number to each logbook, and records the assignment of each logbook distributed (name of person, date distributed, and project number). The purpose of this procedure is to ensure the integrity of the logbook before its use in the field, and to document each logbook assigned to a

project. In the event that more than one logbook is assigned to a project, this process will ensure that all logbooks are accounted for at project closeout.

## 2.0 PROCEDURES

The following subsections provide general guidelines and formatting requirements for field logbooks, and detailed procedures for completing field logbooks.

### 2.1 GENERAL GUIDELINES

- A separate field logbook must be maintained for each project. If a site consists of multiple subsites (or operable units), designate a separate field logbook for each subsite. Similarly, if multiple activities are occurring simultaneously requiring more than one task leader (for example, well installation, private well sampling, or geophysical survey), each task leader should maintain a separate field logbook to ensure that each activity is documented in sufficient detail.

- At larger sites, a general field log may be kept at the site trailer or designated field office to track site visitors, document daily safety meetings, and record overall site issues or occurrences.

- Data from multiple subsites may be entered into one logbook that contains only one type of information for special tasks, such as periodic well water-level measurements.

- All logbooks must be bound and contain consecutively numbered pages. If the pages are not pre-numbered, the sequential page number should be written at the top of each page.

- No pages can be removed from the logbook for any purpose.

- All information must be entered using permanent, waterproof ink, either a traditional ballpoint pen or a permanent marker. Do not use pens with water-based ink (typically identified as rollerball or gel ink pens) because the ink may wash out if the paper gets wet. Pencils are not permissible for field notes because information can be erased. The entries should be written dark enough so that the logbook can be easily photocopied.

- Be sure that all entries are legible. Use print rather than cursive writing and keep the logbook pages free of dirt and moisture to the extent possible.

- Set apart critical information such as sample numbers by circling or drawing a box around the critical data.

- Do not enter information in the logbook that is not related to the project. The language used in the logbook should be factual and objective. Avoid speculation that could conflict with information presented in subsequent project deliverables and correspondence (see Section 1.0 above).

- Use military time, unless otherwise specified by the client. If a logbook entry is not related to a specific event, set it aside with the identification as a “NOTE.”

- Include site sketches, as appropriate.

- Begin a new page for each day’s notes.

- Include the date, project number, and location (if the project has multiple locations) at the top of each page.

- At the end of a day, draw a single diagonal line through any unused lines on the page, and sign at the bottom of the page. Note and implement any client-specific requirements (for example, some clients require each logbook page to be signed).

- Write notes on every line of the logbook. Do not skip any pages or parts of pages unless a day's activity ends in the middle of a page.

- If a line is left blank for some reason, cross it out (with a single line) and initial to prevent unauthorized entries.

- Cross out (with a single line) and initial any edits to the logbook entries. Note and implement any client-specific requirements (for example, some clients also require that edits be dated). Edits should only be made if the initial entry is illegible or erroneous. Do not make corrections for grammar or style.

## 2.2 LOGBOOK FORMAT

The layout and organization of each field logbook should be consistent and generally follow the format guidelines presented below. Some clients or contracts may have specific formatting guidelines that differ somewhat from this SOP; review client requirements at the start of the project to help ensure any client-specific guidelines are integrated.

### 2.2.1 Logbook Cover

Spaces are usually provided on the inside front cover (or the opening page in some logbooks) for the company name, address, contact names, and telephone numbers. If preprinted spaces for this information are not provided in the logbook, write the information on the first available page. Information to be included on the inside front cover or first page includes:

- Logbook document control number (assigned by issuer)
- "Book # of #" (determined by the project manager if there is more than one logbook for the project)
- Contract and task order numbers
- Name of the site and site location (city and state)
- Name of subsite (or operable unit), if applicable
- Type of activity, if the logbook is for a specific activity, such as well installation or indoor air sampling
- Beginning and ending dates of activities entered into the logbook

### 2.2.2 Inside Cover or First Page

Spaces are usually provided on the inside front cover (or the opening page in some logbooks) for the company name, address, contact names, and telephone numbers. If preprinted spaces for this information are not provided in the logbook, write the information on the first available page. Information to be included on the inside front cover or first page includes:

- Tetra Tech project manager and site manager names and telephone numbers
- Tetra Tech office address
- Client contact and telephone number
- Site safety officer and telephone number
- Emergency contact telephone number (911, if applicable, or nearest hospital)
- Subcontractor contacts and telephone numbers
- Site property owner or property manager contact information

Note—some clients prohibit the inclusion of personally identifiable information such as personal mobile telephone numbers on official project records.

## 2.3 ENTERING INFORMATION IN THE LOGBOOK

The following lists provide guidance on the types of information to be included in a typical field logbook. This guidance is general and is not intended to be all-inclusive. Certain projects or clients may specify logbook requirements that are beyond the elements presented in this SOP.

### 2.3.1 General Daily Entries

- Document what time field personnel depart the Tetra Tech office and arrive at the hotel or site. If permitted by the client to charge travel time for site work, document what time personnel leave and arrive at the hotel each day. (This information may be needed at remote sites where hotel accommodations are not near the site.)
- Indicate when all subcontractors arrive and depart the site.
- Note weather conditions at the time of arrival on site and any changes to the weather that might affect completion of project tasks during the day.
- Include the date and project number at the top of each page.
- Document that a site safety meeting was held and include the basic contents of the meeting.
- List the level of personal protection to be used for health and safety.

- Summarize the day's planned activities.
- Summarize which activities each field team member will be doing.

### 2.3.2 Field Activity Entries

- Refer to field data collection forms for details about field data collection activities (for example time, date, depth of samples, and field measurements). If separate field sampling sheets are not used, see Section 2.3.3 regarding logbook entries for sampling activities.
- Refer to well purge forms, well construction logs, and other activity-specific forms as applicable rather than including this type of information in the field logbook. These other forms allow the information to be more accessible at a later date.
- List any air monitoring instrumentation used, with readings and locations.
- Refer to instrument field logs for equipment calibration information.
- Summarize pertinent conversations with site visitors (agency representatives, property owners, client contacts, and local citizens).
- Summarize any problems or deviations from the quality assurance project plan (QAPP) or field sampling plan.
- Document the activities and whereabouts of each team member. (As indicated in Section 2.1, multiple logbooks may be required to ensure sufficient detail for contemporaneous activities).
- Indicate when utility clearances are completed, including which companies participated.
- Indicate when verbal access to a property is obtained.
- Include names, addresses, and telephone numbers of any pertinent site contacts, property owners, and any other relevant personnel.
- Document when lunch breaks or other work stoppages occur.
- Include approximate scale for all diagrams. If a scale is not available, write "not to scale" on the diagram. Indicate the north direction on all maps and cross-sections, and label features on each diagram.

### 2.3.3 Sampling Activity Entries

The following information should typically be on a sample collection log and referenced in the logbook. If the project does not use sample sheets as a result of project-specific requirements, this information should be included in the logbook.

- Location description
- Names of samplers
- Collection time

- Designation of sample as a grab or composite sample
- Identification of blind duplicates or split samples
- Type of sample (water, sediment, soil gas, or other medium)
- On-site measurement data (such as pH, temperature, and specific conductivity)
- Field observations (odors, colors, weather)
- Preliminary sample description
- Type of preservative used
- Instrument readings, if applicable

### 2.3.4 Closing Daily Entries

- Describe decontamination procedures (personnel and equipment).
- Describe handling and disposition of any investigation-derived wastes.
- Summarize which planned activities were completed and which ones were not.
- Note the times that personnel depart the site for the day.
- Summarize any activities conducted after departing the site (paperwork, sample packaging, etc.). This may be required to document billable time incurred after field activities were completed for the day.

### 2.3.5 Photographic Log Entries

- Before using a digital camera, ensure that the system date and time are correct. Verify whether the timestamp is being recorded on the image, if required.
- Indicate in the text that photographs were taken and the location where the photographs can be found (for example, in the project file) and identify the photographer.
- Begin a new photolog page for each new field day.
- Record the time of photograph so that the image can be generally identified when reviewing the digital files.
- Note the direction in which the photograph was taken, along with any relevant details that might not be understood when looking at the photograph.
- In the event that a film camera is used, the sequential number of the image should also be recorded, and the time from the logbook will be the recorded time for the photograph.

## 2.4 LOGBOOK STORAGE

Custody of logbooks must be maintained at all times. During field activities, field personnel must keep the logbooks in a secure place (locked car, trailer, or field office) when the logbook is not in personal possession. When the field work is over, the logbook should be included in the project file, which should be in a secured file cabinet; in addition, if directed by the project manager, scan logbook pages for electronic file management upon returning to the office. The logbook may be referenced in preparing subsequent reports and scanned logbook pages may be included as an appendix to a report. However, it is advisable to obtain direction directly from the client before including the logbook as a report appendix, because its inclusion may not be appropriate in all cases.

## 2.5 HEALTH AND SAFETY CONSIDERATIONS

In addition to the procedures outlined in this SOP, all field staff must be aware of and follow the health and safety practices that result from the Activity Hazard Analyses (AHA) for a project. The AHAs include critical safety procedures, required controls, and minimum personal protective equipment necessary to address potential hazards. The hazards specific to project tasks must be identified and controlled to the extent practicable and communicated to all project personnel via the approved, project-specific health and safety plan.

**SOP APPROVAL FORM**

TETRA TECH, INC.

EMI OPERATING UNIT

LABORATORY ANALYTICAL DATA STANDARD OPERATING PROCEDURE

# LABORATORY ANALYTICAL DATA VERIFICATION – MINIMUM REQUIREMENTS

**SOP NO. 203**

**REVISION NO. 1**

Last Reviewed: January 2019

Quality Assurance Approved

*January 2019*

Date

# 1.0 BACKGROUND

Data quality assurance (QA) is necessary for every project. It is the total integrated process for assuring reliability and defensibility of decisions based on data—including analytical data. In particular, the appropriate level and accurate review of data resulting from chemical and physical analysis are essential to ensure these data are of sufficient quality to support the project's technical requirements.

## 1.1 PURPOSE

The purpose of this standard operating procedure (SOP) is to ensure laboratory data used by Tetra Tech to make project decisions are of the quality required and provide the level of confidence needed to make the appropriate project decisions. This SOP specifies data verification guidelines for ensuring achievement of a minimum level of project data QA.

## 1.2 SCOPE

Analytical data generated for Tetra Tech projects must receive the appropriate level of data review. The level of detail and stringency of data verification or data validation depends on the needs of the project and program. This SOP specifies guidance for data verification procedures when program-specific or regulatory requirements are not defined contractually or by program procedures and regulations (for example, Phase II Environmental Site Assessments, emissions monitoring, and compliance reporting data for permit applications).

## 1.3 DEFINITIONS

This subsection defines key terms used in the text.

**Data package** – A hard copy or electronic report from an analytical laboratory for a set of chemical or physical analyses performed on a group of samples (sometimes referred to as a sample delivery group). The data package should contain sufficient QA documentation to complete data verification and determine data usability (as discussed in Section 1.5 of this SOP).

**Data usability** – A qualitative decision process whereby a qualified person determines whether the data may be used for the intended purpose. Data should be classified into one of the following two categories: usable or rejected (unusable).

**Data verification** – The act of determining and documenting whether data conform to specified requirements. The determination may involve processes such as reviewing, inspecting, testing, checking, recalculating, and auditing (EPA 2002).

**Rejected data** – Data that do not conform to some or all requirements considered critical to assuring and confirming the quality of the data. Nonconformances may include: (1) critical quality control (QC) criteria are not met (see Table 1); (2) appropriate methods were not followed or the methods used involved significant deviations that might impact data quality or meaning; and (3) critical documentation is missing or incomplete.

**Sample delivery group** – A unit (group) of samples received by the laboratory during a field sampling event. A "sample delivery group" (SDG) is typically composed of 20 or fewer samples and is grouped based on the number of samples and not the analytical testing requested. An SDG may be defined based on the number of samples received by the laboratory on a given day or over a period of up to 7 calendar days.

**Qualified person** – A chemist or other person who has received training in or has demonstrated skills and knowledge of laboratory procedures and QC. The qualified person involved in data verification should understand the data generation procedures and know project documentation and data quality requirements. Although data validation is beyond the scope of this SOP, a qualified person should be capable of providing the necessary level of professional judgment (which requires familiarity with data validation procedures). Examples of data validation guidance can be found in EPA's *National Functional Guidelines for Superfund Methods Data Review* (EPA 2016, 2017a, 2017b), though some projects may rely on guidance from other sources.

**Usable data** – Data conforming to most or all requirements considered critical to assuring and confirming the quality of the data. Conformances important to achieve usability include: (1) critical QC criteria are met (see Table 1); (2) appropriate methods were followed, or only minor deviations to the methods were made that would not impact data quality or meaning; and (3) critical documentation is complete. Professional judgment by a qualified person should be used to determine data usability.

## 1.4 REFERENCES

U.S. Environmental Protection Agency (EPA). 2002. *Guidance on Environmental Data Verification and Data Validation (EPA QA/G-8)*. Office of Environmental Information. Washington, DC. EPA/240/R-02/004. November. Reissued January 2008. On-line address: https://www.epa.gov/sites/production/files/2015-06/documents/g8-final.pdf

EPA. 2016. *National Functional Guidelines for High Resolution Superfund Methods Data Review*. Office of Superfund Remediation and Technology Innovation (OSRTI). Washington, DC. EPA-542-B-16-001. April. On-line address: https://www.epa.gov/sites/production/files/2016-05/documents/hrsm_nfg.pdf

EPA. 2017a. *National Functional Guidelines for Organic Superfund Methods Data Review*. OSRTI. Washington, DC. EPA-540-R-2017-002. January. On-line address: https://www.epa.gov/sites/production/files/2017-01/documents/national_functional_guidelines_for_organic_superfund_methods_data_review_013072017.pdf

EPA. 2017b. *National Functional Guidelines for Inorganic Superfund Methods Data Review*. OSRTI. Washington, DC. EPA-540-R-2017-001. January. On-line address: https://www.epa.gov/sites/production/files/2017-01/documents/national_functional_guidelines_for_inorganic_superfund_methods_data_review_01302017.pdf

## 1.5 REQUIREMENTS AND RESOURCES

The following are required for laboratory data verification as described in this SOP:

- Laboratory data package(s)
- Project-specific information for data use (that is, work plan, sampling and analysis plan [SAP], quality assurance project plan [QAPP], proposal, or purchase order)
- Qualified person, familiar with laboratory procedures and capable of determining data usability.

Laboratory data package(s) should include the following to allow for data verification:

- Cover letter or case narrative, including the laboratory name and address, that certifies analytical results via signature of the project chemist, QA manager, or laboratory manager
- Signed field chain-of-custody form(s)
- Sample receipt and log-in forms, which include general comments and specify temperature, holding time, bottle breakages, and any nonconformances or discrepancies (Note: this information is sometimes included on the chain-of-custody form)
- Laboratory log-in summary, including laboratory sample ID, field sample ID, list of analyses performed, and analytical methods employed (Note: this information is occasionally included on the analytical results forms and not on a separate summary)
- Analytical results
- Applicable analytical batch QC results (for example, method and field blanks, surrogate spikes, matrix spike/matrix spike duplicates [MS/MSD], and laboratory control sample/laboratory control sample duplicates [LCS/LCSD])
- List of laboratory data qualifier definitions.

Time required for laboratory data verification can vary greatly depending on the number and type of analyses per data package, and the number of samples per data package. The following rules of thumb, including producing a record of the type found in Attachment A, may be useful for planning purposes:

- 30 minutes for an SDG with one major analysis (for example, metals or volatiles)
- 90 minutes to 2 hours for an SDG with a common suite of analyses (for example, metals, volatiles, semivolatiles, pesticides, polychlorinated biphenyls, and total petroleum hydrocarbons)
- 30 minutes for an SDG with a common suite of wet chemistry analyses (for example, alkalinity, pH, major anions, total organic carbon, total dissolved solids, and total suspended solids).

The times noted are estimates only. Involving a qualified person in the planning process will help ensure proper budget for data verification.

## 2.0 PROCEDURES

***Step 1*** – The project manager identifies a qualified person with an understanding of laboratory data generation and usability to review and verify the data. If the data are released to the client prior to verification, the client should be advised that the data are preliminary pending this review. It is possible that more than one qualified person may be assigned; for example, one person to review the samples analyzed by the laboratory for completeness against the SAP, and another to verify the data quality.

***Step 2*** – The qualified person identifies the project analytical QA/QC needs for documentation and technical specifications as these apply to data content and quality. A work plan, SAP, QAPP, regulatory guidance, laboratory analytical method, client contract, or project scope of work may identify the technical specifications and QA/QC requirements.

***Step 3*** – The qualified person reviews the data and documents the review findings based on the requirements for data quality needed to achieve project objectives. Serious issues regarding data usability are immediately brought to the project manager's attention for further discussion and resolution. Table 1 describes the elements of data verification.

In all cases, the laboratory chain-of-custody form indicating sample IDs, matrices, and analytical methods—and perhaps frequency of collection and submittal of QA/QC samples (such as field duplicates, trip blanks, field blanks, equipment rinsate blanks, and MS/MSDs)—should be cross-checked with the SAP or the contracted scope of work.

In each case, professional judgment should be used to determine data usability. Ultimately, the project manager's responsibility is to ensure a qualified person has reviewed the laboratory data package and has deemed the data usable for the data's intended purpose.

***Step 4*** – The qualified person reviews and compares the analytical method detection limits (MDL), reporting limits (RL), and practical quantitation limits (PQL) for compliance with project requirements. Explicit definition and clarification of MDLs, RLs, and PQLs should be established before field activities begin.

***Step 5*** – The qualified person communicates findings. The deliverable from the qualified person includes at least one of the following:

- An e-mail indicating data usability
- A memorandum summarizing the evaluated results
- A table of data showing data considered biased or outside acceptance criteria for various data quality indicators by a large enough factor that use of the data might affect environmental decisions.

Some written form of communication should be provided for the project file. An example of a minimum data verification deliverable is included as Attachment A.

## 3.0 DATA VERIFICATION RESULTS

As described above, potential data verification issues involving the following designations may be encountered during this process:

**Rejected data** – During verification, the qualified person may reject some or all of the data (that is, consider the data unusable). If laboratory data are rejected based on poor quality, the project manager may ask the laboratory to re-analyze the extract, or re-digest and re-extract the original sample if enough volume remains. If reanalysis or re-digestion and re-extraction of samples are not viable options, re-collection of the samples may be required.

**Inadequate data** – The qualified person may find the data inadequate for the intended purpose, even if all QC criteria were met—for example, a case in which laboratory reporting limits are not adequate to meet the comparison or screening values established during the project planning process.

**Incomplete data packages** – The data package provided by the laboratory may not be complete. If the laboratory data package does not include the minimum contents defined in Section 1.5, the laboratory should be notified and required to issue a revised data package.

If any of the above data designations are assigned by the data verifier, the situation should be addressed immediately and corrected to minimize effects on future project deliverables. Further discussion with the analytical laboratory may help in the effort to address each of the above situations. The data verifier and the project manager should discuss potential remedies or corrective measures to minimize impact(s) of the above situations on project analytical data and decisions based on those data.

**Table 1**
**Elements of Laboratory Data Verification**

| Data Report Element | Minimum Required Review | Actions |
|---|---|---|
| Chain-of-custody form | Review laboratory log-in information against chain-of-custody forms and the contracted scope of work or planning documents for: accuracy and completeness of documentation, sample quantity and IDs, proper signatures attesting to chain of custody, sample condition upon receipt (breakage, temperature, etc.), sample preservation (see below), and analytical method selection. | Discrepancies regarding log-in, chain of custody, analytical method selection, or related issues should be immediately addressed. If discrepancies are identified, the laboratory should be contacted immediately, and corrective actions implemented. Improper sample handling and preservation should be investigated to determine sample adequacy (see "Sample preservation, storage, and holding times" below). |
| Data package completeness | Review data package to make sure that all requested analytical procedures have occurred and required corresponding data are reported. | Analytical results that lack supporting data and information may be considered invalid and not usable for the purpose intended. Such conditions should be immediately addressed with the project team and laboratory. |
| Sample preservation, storage, and holding times | Review sample preservation, storage, and holding times in compliance with selected analytical method and matrix. | Analytical results of samples not properly preserved and stored or digested/extracted or analyzed outside the appropriate holding time, may be considered invalid and not usable for the purpose intended. Such conditions should be immediately addressed with the project team. |
| Method and field blanks | Review blank data for positive results that may indicate possible field or laboratory contamination. | If blank contamination is found in either the laboratory method blanks or the field QC blanks (that is, equipment rinsate blanks, source or field blanks, or trip blanks), associated sample results should be reviewed. Detections in the associated environmental samples may be attributed to laboratory or field contamination, and qualifications of the data may be necessary. |
| Precision and accuracy* (may include surrogate spikes, MS/MSDs, and LCS/LCSDs, as well as additional QC elements) | Review QC data summaries for the analytical method used. Use project-required, method-required, or laboratory-provided control limits. Review laboratory-assigned data quality flags and notations, and revise if necessary. | In general, recoveries and relative percent difference values for surrogate spikes, MS/MSDs, LCS/LCSDs, or other reviewed QC elements that fall outside of the specified control limits may indicate problems with the laboratory analysis.* |

**Notes:**

| | |
|---|---|
| * | The type and amount of QC information available for review will depend upon the analytical method and level of data package requested. |
| QC | Quality control |
| LCS/LCSD | Laboratory control sample/laboratory control sample duplicate |
| MS/MSD | Matrix spike/matrix spike duplicate |

**ATTACHMENT A**
**EXAMPLE DATA VERIFICATION REPORT**

**Prepared by:** _____

**Date:** _____

**Site Name/Job Number:** _____

**Laboratory:** _____

**Data Package or SDG Number:** _____

**Sample Designations/Names (ID):** _____

**Matrices:** _____

**Analytical Parameters:** _____

| Data Package Element | Usable | Rejected | NA | Description of Affected Data (note specific samples and analytical parameters affected) |
|---|---|---|---|---|
| Chain-of-custody form | | | | |
| Data package completeness | | | | |
| Sample preservation, storage, and holding times | | | | |
| Method and field blank contamination | | | | |
| Surrogate spikes | | | | |
| Matrix spikes/matrix spike duplicates (MS/MSD) | | | | |
| Laboratory control samples/laboratory control sample duplicates (LCS/LCSD) | | | | |
| Other | | | | |
| Summary | | | | |

**Notes:**

NA Not applicable
SDG Sample delivery group

**ATTACHMENT 1**

**ADDITIONAL STANDARD OPERATING PROCEDURES (SOP)**

| **SOP NO.** | **TITLE** |
|---|---|
| ERT SOP 2015 | Asbestos Sampling |
| REAC SOP 1025 | Data Verification/Validation for Procedures for Asbestos in Air by TEM Analysis (Based on ISO 10312) |

## ASBESTOS SAMPLING

CONTENTS

CONTENTS (cont)

# ASBESTOS SAMPLING

## 1.0 SCOPE AND APPLICATION

Asbestos has been used in many commercial products including building materials such as flooring tiles and sheet goods, paints and coatings, insulation, and roofing asphalts. These products and others may be found at hazardous waste sites hanging on overhead pipes, contained in drums, abandoned in piles, or as part of a structure. Asbestos tailing piles from mining operations can also be a source of ambient asbestos fibers. Asbestos is a known carcinogen and requires air sampling to assess airborne exposure to human health. This Standard Operating Procedure (SOP) provides procedures for asbestos air sampling by drawing a known volume of air through a mixed cellulose ester (MCE) filter. The filter is then sent to a laboratory for analysis. The U.S. Environmental Protection Agency/Environmental Response Team (U.S. EPA/ERT) uses one of four analytical methods for determining asbestos in air. These include: U.S. EPA's Environmental Asbestos Assessment Manual, Superfund Method for the Determination of Asbestos in Ambient Air for Transmission Electron Microscopy (TEM)[1]; U.S. EPA's Modified Yamate Method for TEM[2]; National Institute for Occupational Safety and Health (NIOSH) Method 7402 (direct method only) for TEM; and NIOSH Method 7400 for Phase Contrast Microscopy (PCM)[3]. Each method has specific sampling and analytical requirements (i.e., sample volume and flow rate) for determining asbestos in air.

The U.S. EPA/ERT typically follows procedures outlined in the TEM methods for determining mineralogical types of asbestos in air and for distinguishing asbestos from non-asbestos minerals. The Phase Contrast Microscopy (PCM) method is used by U.S. EPA/ERT as a screening tool since it is less costly than TEM. PCM cannot distinguish asbestos from non-asbestos fibers, therefore the TEM method may be necessary to confirm analytical results. For example, if an action level for the presence of fibers has been set and PCM analysis indicates that the action level has been exceeded, then TEM analysis can be used to quantify and identify asbestos structures through examination of their morphology crystal structures (through electron diffraction), and elemental composition (through energy dispersive X-ray analysis). In this instance samples should be collected for both analyses in side by side sampling trains (some laboratories are able to perform PCM and TEM analysis from the same filter). The Superfund method is designed specifically to provide results suitable for supporting risk assessments at Superfund sites, it is applicable to a wide range of ambient air situations at hazardous waste sites. U.S. EPA's Modified Yamate Method for TEM is also used for ambient air sampling due to high volume requirements. The PCM and TEM NIOSH analytical methods require lower sample volumes and are typically used indoors; however, ERT will increase the volume requirement for outdoor application.

Other Regulations pertaining to asbestos have been promulgated by U.S. EPA and OSHA. U.S. EPA's National Emission Standards for Hazardous Air Pollutants (NESHAP) regulates asbestos-containing waste materials. NESHAP establishes management practices and standards for the handling of asbestos and emissions from waste disposal operations (40 CFR Part 61, Subparts A and M). U.S. EPA's 40 CFR 763 (July 1, 1987)[4] and its addendum 40 CFR 763 (October 30, 1987)[4] provide comprehensive rules for the asbestos abatement industry. State and local regulations on these issues vary and may be more stringent than federal requirements. The OSHA regulations in 29 CFR 1910.1001 and 29 CFR 1926.58 specify work practices and safety equipment such as respiratory protection and protective clothing when handling asbestos. The OSHA standard for an 8-hour, time-weighted average (TWA) is 0.2 fibers/cubic centimeters of air. This standard pertains to fibers with a length-to-width ratio of 3 to 1 with a fiber length >5 µm[5,6]. An action level of 0.1 fiber/cc (one-half the OSHA standard) is the level U.S. EPA has established in which employers must initiate such activities as air monitoring, employee training, and medical surveillance[5,6].

These are standard (i.e., typically applicable) operating procedures which may be varied or changed as required, dependent upon site conditions, equipment limitations or limitations imposed by the procedure. In all instances, the ultimate procedures employed should be documented and associated with the final report.

Mention of trade names or commercial products does not constitute U.S. EPA endorsement or recommendation for use.

## 2.0 METHOD SUMMARY

Prior to sampling, the site should be characterized by identifying on-site as well as off-site sources of airborne asbestos. The array of sampling locations and the schedule for sample collection is critical to the success of an investigation. Generally, sampling strategies to characterize a single point source are fairly straightforward, while multiple point sources and area sources increase the complexity of the sampling strategy. It is not within the scope of this SOP to provide a generic asbestos air sampling plan. Experience, objectives, and site characteristics will dictate the sampling strategy.

During a site investigation, sampling stations should be arranged to distinguish spatial trends in airborne asbestos concentrations. Sampling schedules should be fashioned to establish temporal trends. The sampling strategy typically requires that the concentration of asbestos at the source (worst case) or area of concern (downwind), crosswind, as well as background (upwind) contributions be quantified. See Table 1 (Appendix A) for U.S. EPA/ERT recommended sampling set up for ambient air. Indoor asbestos sampling requires a different type of strategy which is identified in Table 2 (Appendix A). It is important to establish background levels of contaminants in order to develop a reference point from which to evaluate the source data. Field blanks and lot blanks can be utilized to determine other sources.

Much information can be derived from each analytical method previously mentioned. Each analytical method has specific sampling requirements and produce results which may or may not be applicable to a specific sampling effort. The site sampling objectives should be carefully identified so as to select the most appropriate analytical method. Additionally, some preparation (i.e., lot blanks results) prior to site sampling may be required, these requirements are specified in the analytical methods.

## 3.0 SAMPLE PRESERVATION, CONTAINERS, HANDLING, AND STORAGE

### 3.1 Sample Preservation

No preservation is required for asbestos samples.

### 3.2 Sample Handling, Container and Storage Procedures

1. Place a sample label on the cassette indicating a unique sampling number. Do not put sampling cassettes in shirt or coat pockets as the filter can pick up fibers. The original cassette box is used to hold the samples.

2. Wrap the cassette individually in a plastic sample bag. Each bag should be marked indicating sample identification number, total volume, and date.

3. The wrapped sampling cassettes should be placed upright in a rigid container so that the cassette cap is on top and cassette base is on bottom. Use enough packing material to prevent jostling or damage. Do not use vermiculite as packing material for samples. If possible, hand carry to lab.

4. Provide appropriate documentation with samples (i.e., chain of custody and requested analytical methodology).

## 4.0 INTERFERENCES AND POTENTIAL PROBLEMS

Flow rates exceeding 16 liters/minute (L/min) which could result in filter destruction due to (a) failure of its physical support under force from the increased pressure drop; (b) leakage of air around the filter mount so that the filter is bypassed, or (c) damage to the asbestos structures due to increased impact velocities.

### 4.1 U.S. EPA's Superfund Method

#### 4.1.1 Direct-transfer TEM Specimen Preparation Methods

Direct-Transfer TEM specimen preparation methods have the following significant interferences:

- The achievable detection limit is restricted by the particulate density on the filter, which in turn is controlled by the sampled air volume and the total suspended particulate concentration in the atmosphere being sampled.

- The precision of the result is dependent on the uniformity of the deposit of asbestos structures on the sample collection filter.

- Air samples must be collected so that they have particulate and fiber loadings within narrow ranges. If too high a particulate loading occurs on the filter, it is not possible to prepare satisfactory TEM specimens by a direct-transfer method. If too high a fiber loading occurs on the filter, even if satisfactory TEM specimens can be prepared, accurate fiber counting will not be possible.

#### 4.1.2 Indirect TEM Specimen Preparation Methods

Indirect TEM specimen preparation methods have the following interferences:

- The size distribution of asbestos structures is modified.

- There is increased opportunity for fiber loss or introduction of extraneous contamination.

- When sample collection filters are ashed, any fiber contamination in the filter medium is concentrated on the TEM specimen grid.

It can be argued that direct methods yield an under-estimate of the asbestos structure concentration because many of the asbestos fibers present are concealed by other particulate material with which they are associated. Conversely, indirect methods can be considered to yield an over-estimate because some types of complex asbestos structures disintegrate during the preparation, resulting in an increase in the numbers of structures counted.

4.2 U.S. EPA's Modified Yamate Method for TEM

High concentrations of background dust interfere with fiber identification.

4.3 NIOSH Method for TEM

Other amphibole particles that have aspect ratios greater than 3:1 and elemental compositions similar to the asbestos minerals may interfere in the TEM analysis. Some non-amphibole minerals may give electron diffraction patterns similar to amphiboles. High concentrations of background dust interfere with fiber identification.

4.4 NIOSH Method for PCM

PCM cannot distinguish asbestos from non-asbestos fibers; therefore, all particles meeting the counting criteria are counted as total asbestos fibers. Fiber less than 0.25 um in length will not be detected by this method. High levels of non-fibrous dust particles may obscure fibers in the field of view and increase the detection limit.

5.0 EQUIPMENT/MATERIALS

5.1 Sampling Pump

The constant flow or critical orifice controlled sampling pump should be capable of a flow-rate and pumping time sufficient to achieve the desired volume of air sampled.

The lower flow personal sampling pumps generally provide a flow rate of 20 cubic centimeters/minute (cc/min) to 4 L/min. These pumps are usually battery powered. High flow pumps are utilized when flow rates between 2 L/min to 20 L/min are required. High flow pumps are used for short sampling periods so as to obtain the desired sample volume. High flow pumps usually run on AC power and can be plugged into a nearby outlet. If an outlet is not available then a generator should be obtained. The generator should be positioned downwind from the sampling pump. Additional voltage may be required if more than one pump is plugged into the same generator. Several electrical extension cords may be required if sampling locations are remote.

The recommended volume for the Superfund method (Phase I) requires approximately 20 hours to collect. Such pumps typically draw 6 amps at full power so that 2 lead/acid batteries should provide sufficient power to collect a full sample. The use of line voltage, where available, eliminates the difficulties associated with transporting stored electrical energy.

A stand should be used to hold the filter cassette at the desired height for sampling and the filter

cassette shall be isolated from the vibrations of the pump.

5.2 Filter Cassette

The cassettes are purchased with the required filters in position, or can be assembled in a laminar flow hood or clean area. When the filters are in position, a shrink cellulose band or adhesive tape should be applied to cassette joints to prevent air leakage.

5.2.1 TEM Cassette Requirements

Commercially available field monitors, comprising 25 mm diameter three-piece cassettes, with conductive extension cowls shall be used for sample collection. The cassette must be new and not previously used. The cassette shall be loaded with an MCE filter of pore size 0.45 µm, and supplied from a lot number which has been qualified as low background for asbestos determination. The cowls should be constructed of electrically conducting material to minimize electrostatic effects. The filter shall be backed by a 5 µm pore size MCE filter (Figure 1, Appendix B).

5.2.2 PCM Cassette Requirements

NIOSH Method 7400, PCM involves using a 0.8 to 1.2 µm mixed cellulose ester membrane, 25 mm diameter, 50 mm conductive cowl on cassette (Figure 2, Appendix B). Some labs are able to perform PCM and TEM analysis on the same filter; however, this should be discussed with the laboratory prior to sampling.

5.3 Other Equipment

- Inert tubing with glass cyclone and hose barb
- Whirlbags (plastic bags) for cassettes
- Tools - small screw drivers
- Container - to keep samples upright
- Generator or electrical outlet (may not be required)
- Extension cords (may not be required)
- Multiple plug outlet
- Sample labels
- Air data sheets
- Chain of Custody records

6.0 REAGENTS

Reagents are not required for the preservation of asbestos samples.

7.0 PROCEDURES

7.1 Air Volumes and Flow Rates

Sampling volumes are determined on the basis of how many fibers need to be collected for reliable

measurements. Therefore, one must estimate how many airborne fibers may be in the sampling location.

Since the concentration of airborne aerosol contaminants will have some effect on the sample, the following is a suggested criterion to assist in selecting a flow rate based on real-time aerosol monitor (RAM) readings in milligrams/cubic meter ($mg/m^3$).

| | Concentration | Flow Rate |
|---|---|---|
| • Low RAM readings: | <6.0 $mg/m^3$ | 11-15 L/min |
| • Medium RAM readings | >6.0 $mg/m^3$ | 7.5 L/min |
| • High RAM readings: | >10. $mg/m^3$ | 2.5 L/min |

In practice, pumps that are available for environmental sampling at remote locations operate under a maximum load of approximately 12 L/min.

7.1.1 U.S. EPA's Superfund Method

The Superfund Method incorporates an indirect preparation procedure to provide flexibility in the amount of deposit that be can be tolerated on the sample filter and to allow for the selective concentration of asbestos prior to analysis. To minimize contributions to background contamination from asbestos present in the plastic matrices of membrane filters while allowing for sufficient quantities of asbestos to be collected, this method also requires the collection of a larger volume of air per unit area of filter than has traditionally been collected for asbestos analysis. Due to the need to collect large volumes of air, higher sampling flow rates are recommended in this method than have generally been employed for asbestos sampling in the past. As an alternative, samples may be collected over longer time intervals. However, this restricts the flexibility required to allow samples to be collected while uniform meteorological conditions prevail.

The sampling rate and the period of sampling should be selected to yield as high a sampled volume as possible, which will minimize the influence of filter contamination. Wherever possible, a volume of 15 cubic meters (15,000 L) shall be sampled for those samples intended for analysis only by the indirect TEM preparation method (Phase 1 samples). For those samples to be prepared by both the indirect and the direct specimen preparation methods (Phase 2 samples), the volumes must be adjusted so as to provide a suitably-loaded filter for the direct TEM preparation method. One option is to collect filters at several loadings to bracket the estimated optimum loading for a particular site. Such filters can be screened in the laboratory so that only those filters closest to optimal loading are analyzed. It has been found that the volume cannot normally exceed 5 cubic meters (5000 L) in an urban or agricultural area, and 10 cubic meters (10,000 L) in a rural area for samples collected on a 25 mm filter and prepared by a direct-transfer technique.

An upper limit to the range of acceptable flow rates for this method is 15 L/min. At many locations, wind patterns exhibit strong diurnal variations. Therefore, intermittent sampling (sampling over a fixed time interval repeated over several days) may be

necessary to accumulate 20 hours of sampling time over constant wind conditions. Other sampling objectives also may necessitate intermittent sampling. The objective is to design a sampling schedule so that samples are collected under uniform conditions throughout the sampling interval. This method provides for such options. Air volumes collected on Phase I samples are maximized (<16 L/min). Air volumes collected on Phase 2 samples are limited to provide optimum loading for filters to be prepared by a direct-transfer procedure.

7.1.2 U.S. EPA's Modified Yamate Method for TEM

U.S. EPA's TEM method requires a minimum volume of 560 L and a maximum volume of 3,800 L in order to obtain an analytical sensitivity of 0.005 structures/cc. The optimal volume for TEM is 1200 L to 1800 L. These volumes are determined using a 200 mesh EM grid opening with a 25-mm filter cassette. Changes in volume would be necessary if a 37-mm filter cassette is used since the effective area of a 25 mm (385 sq mm) and 37 mm (855 sq m) differ.

7.1.3 NIOSH Method for TEM and PCM

The minimum recommended volume for TEM and PCM is 400 L at 0.1 fiber/cc. Sampling time is adjusted to obtain optimum fiber loading on the filter. A sampling rate of 1 to 4 L/min for eight hours (700 to 2800 L) is appropriate in non-dusty atmospheres containing 0.1 fiber/cc. Dusty atmospheres i.e., areas with high levels of asbestos, require smaller sample volumes (<400 L) to obtain countable samples.

In such cases, take short, consecutive samples and average the results over the total collection time. For documenting episodic exposures, use high flow rates (7 to 16 L/min) over shorter sampling times. In relatively clean atmospheres where targeted fiber concentrations are much less than 0.1 fiber/cc, use larger sample volumes (3,000 to 10,000 L) to achieve quantifiable loadings. Take care, however, not to overload the filter with background dust. If > 50% of the filter surface is covered with particles, the filter may be too overloaded to count and will bias the measured fiber concentration. Do not exceed 0.5 mg total dust loading on the filter.

7.2 Calibration Procedures

In order to determine if a sampling pump is measuring the flow rate or volume of air correctly, it is necessary to calibrate the instrument. Sampling pumps should be calibrated immediately before and after each use. Preliminary calibration should be conducted using a primary calibrator such as a soap bubble type calibrator, (e.g., a Buck Calibrator, Gilibrator, or equivalent primary calibrator) with a representative filter cassette installed between the pump and the calibrator. The representative sampling cassette can be reused for calibrating other pumps that will be used for asbestos sampling. The same cassette lot used for sampling should also be used for the calibration. A sticker should be affixed to the outside of the extension cowl marked "Calibration Cassette."

A rotameter can be used provided it has been recently pre-calibrated with a primary calibrator.

Three separate constant flow calibration readings should be obtained both before sampling and after sampling. Should the flow rate change by more than 5% during the sampling period, the average of the pre- and post-calibration rates will be used to calculate the total sample volume. The sampling pump used shall provide a non-fluctuating air-flow through the filter, and shall maintain the initial volume flow-rate to within " 10% throughout the sampling period. The mean value of these flow-rate measurements shall be used to calculate the total air volume sampled. A constant flow or critical orifice controlled pump meets these requirements. If at any time the measurement indicates that the flow-rate has decreased by more than 30%, the sampling shall be terminated. Flexible tubing is used to connect the filter cassette to the sampling pump Sampling pumps can be calibrated prior to coming on-site so that time is saved when performing on-site calibration.

7.2.1 Calibrating a Personal Sampling Pump with an Electronic Calibrator

1. See manufacturer's manual for operational instructions.

2. Set up the calibration train as shown in (Figure 3, Appendix B) using a sampling pump, electronic calibrator, and a representative filter cassette. The same lot sampling cassette used for sampling should also be used for calibrating.

3. To set up the calibration train, attach one end of the PVC tubing (approx. 2 foot) to the cassette base; attach the other end of the tubing to the inlet plug on the pump. Another piece of tubing is attached from the cassette cap to the electronic calibrator.

4. Turn the electronic calibrator and sampling pump on. Create a bubble at the bottom of the flow chamber by pressing the bubble initiate button. The bubble should rise to the top of the flow chamber. After the bubble runs its course, the flow rate is shown on the LED display.

5. Turn the flow adjust screw or knob on the pump until the desired flow rate is attained.

6. Perform the calibration three times until the desired flow rate of " 5% is attained.

7.2.2 Calibrating a Rotameter with an Electronic Calibrator

1. See manufacturer's manual for operational instructions.

2. Set up the calibration train as shown in (Figure 4, Appendix B) using a sampling pump, rotameter, and electronic calibrator.

3. Assemble the base of the flow meter with the screw provided and tighten in place. The flow meter should be mounted within $6^{o}$ vertical.

4. Turn the electronic calibrator and sampling pump on.

5. Create a bubble at the bottom of the flow chamber by pressing the bubble initiate button. The bubble should rise to the top of the flow chamber. After the bubble runs its course, the flow rate is shown on the LED display.

6. Turn the flow adjust screw or knob on the pump until the desired flow rate is attained.

7. Record the electronic calibrator flow rate reading and the corresponding rotameter reading. Indicate these values on the rotameter (sticker). The rotameter should be able to work within the desired flow range. Readings can also be calibrated for 10 $cm^3$ increments for Low Flow rotameters, 500 $cm^3$ increments for medium flow rotameters and 1 liter increments for high flow rotameters.

8. Perform the calibration three times until the desired flow rate of " 5% is attained. Once on site, a secondary calibrator, i.e., rotameter may be used to calibrate sampling pumps.

7.2.3 Calibrating a Personal Sampling Pump with a Rotameter

1. See manufacturer's manual for Rotameter's Operational Instructions.

2. Set up the calibration train as shown in (Figure 5, Appendix B) using a rotameter, sampling pump, and a representative sampling cassette.

3. To set up the calibration train, attach one end of the PVC tubing (approx. 2 ft) to the cassette base; attach the other end of the tubing to the inlet plug on the pump. Another piece of tubing is attached from the cassette cap to the rotameter.

4. Assemble the base of the flow meter with the screw provided and tighten in place. The flow meter should be mounted within $6^o$ vertical.

5. Turn the sampling pump on.

6. Turn the flow adjust screw (or knob) on the personal sampling pump until the float ball on the rotameter is lined up with the pre-calibrated flow rate value. A sticker on the rotameter should indicate this value.

7. A verification of calibration is generally performed on-site in the clean zone immediately prior to the sampling.

7.3. Meteorology

It is recommended that a meteorological station be established. If possible, sample after two to three days of dry weather and when the wind conditions are at 10 mph or greater. Record wind speed, wind direction, temperature, and pressure in a field logbook. Wind direction is particularly

important when monitoring for asbestos downwind from a fixed source.

7.4 Ambient Sampling Procedures

7.4.1 Pre-site Sampling Preparation

1. Determine the extent of the sampling effort, the sampling methods to be employed, and the types and amounts of equipment and supplies needed.

2. Obtain necessary sampling equipment and ensure it is in working order and fully charged (if necessary).

3. Perform a general site survey prior to site entry in accordance with the site specific Health and Safety plan.

4. Once on-site the calibration is performed in the clean zone. The calibration procedures are listed in Section 7.2.

5. After calibrating the sampling pump, mobilize to the sampling location.

7.4.2 Site Sampling

1. To set up the sampling train, attach the air intake hose to the cassette base. Remove the cassette cap (Figure 6 and 7, Appendix B). The cassette should be positioned downward, perpendicular to the wind

2. If AC or DC electricity is required then turn it on. If used, the generator should be placed 10 ft. downwind from the sampling pump.

3. Record the following in a field logbook: date, time, location, sample identification number, pump number, flow rate, and cumulative time.

4. Turn the pump on. Should intermittent sampling be required, sampling filters must be covered between active periods of sampling. To cover the sample filter: turn the cassette to face upward, place the cassette cap on the cassette, remove the inlet plug from the cassette cap, attach a rotameter to the inlet opening of the cassette cap to measure the flow rate, turn off the sampling pump, place the inlet plug into the inlet opening on the cassette cap. To resume sampling: remove the inlet plug, turn on the sampling pump, attach a rotameter to measure the flow rate, remove the cassette cap, replace the inlet plug in the cassette cap and invert the cassette, face downward and perpendicular to the wind.

5. Check the pump at sampling midpoint if sampling is longer than 4 hours. The generators may need to be regased depending on tank size. If a filter darkens in appearance or if loose dust is seen in the filter, a second sample should be started.

6. At the end of the sampling period, orient the cassette up, turn the pump off.

7. Check the flow rate as shown in Section 7.2.3. When sampling open-faced, the sampling cap should be replaced before post calibrating. Use the same cassette used for sampling for post calibration (increase dust/fiber loading may have altered the flow rate.

8. Record the post flow rate.

9. Record the cumulative time or run.

10. Remove the tubing from the sampling cassette. Still holding the cassette upright, replace the inlet plug on the cassette cap and the outlet plug on the cassette base.

7.4.3. Post Site Sampling

1. Follow handling procedures in Section 3.2 steps 1-4.

2. Obtain an electronic or hard copy of meteorological data which occurred during the sampling event. Record weather: wind speed, ambient temperature, wind direction, and precipitation. Obtaining weather data several days prior to the sampling event can also be useful.

7.5 Indoor Sampling Procedures

PCM analysis is used for indoor air samples. When analysis shows total fiber count above the OSHA action level 0.1 f/cc then TEM (U.S. EPA's Modified Yamate Method) is used to identify asbestos from non-asbestos fibers.

Sampling pumps should be placed four to five feet above ground level away from obstructions that may influence air flow. The pump can be placed on a table or counter. Refer to Table 2 (Appendix A) for a summary of indoor sampling locations and rationale for selection.

Indoor sampling utilizes high flow rates to increased sample volumes (2000 L for PCM and 2800 to 4200 L for TEM) in order to obtain lower detection limits below the standard, (i.e., 0.01 f/cc or lower [PCM] and 0.005 structures/cc or lower [TEM]).

7.5.1 Aggressive Sampling Procedures

Sampling equipment at fixed locations may fail to detect the presence of asbestos fibers. Due to limited air movement, many fibers may settle out of the air onto the floor and other surfaces and may not be captured on the filter. In the past, an 8-hour sampling period was recommended to cover various air circulation conditions. A quicker and more effective way to capture asbestos fibers is to circulate the air artificially so that the fibers remain airborne during sampling. The result from this sampling option typifies worst case condition. This is referred to as aggressive air sampling for asbestos. Refer to Table

2 for sample station locations.

1. Before starting the sampling pumps, direct forced air (such as a 1-horsepower leaf blower or large fan) against walls, ceilings, floors, ledges, and other surfaces in the room to initially dislodge fibers from surfaces. This should take at least 5 minutes per 1000 sq. ft. of floor.

2. Place a 20-inch fan in the center of the room. (Use one fan per 10,000 cubic feet of room space.) Place the fan on slow speed and point it toward the ceiling.

3. Follow procedures in Section 7.4.1 and 7.4.2 (Turn off the pump and then the fan(s) when sampling is complete.).

4. Follow handling procedures in Section 3.2 steps 1-4.

## 8.0 CALCULATIONS

The sample volume is calculated from the average flow rate of the pump multiplied by the number of minutes the pump was running (volume = flow rate X time in minutes). The sample volume should be submitted to the laboratory and identified on the chain of custody for each sample (zero for lot, field and trip blanks).

The concentration result is calculated using the sample volume and the numbers of asbestos structures reported after the application of the cluster and matrix counting criteria.

## 9.0 QUALITY ASSURANCE/QUALITY CONTROL

Follow all QA/QC requirements from the laboratories as well as the analytical methods.

### 9.1 TEM Requirements

1. Examine lot blanks to determine the background asbestos structure concentration.

2. Examine field blanks to determine whether there is contamination by extraneous asbestos structures during specimen preparation.

3. Examine of laboratory blanks to determine if contamination is being introduced during critical phases of the laboratory program.

4. To determine if the laboratory can satisfactorily analyze samples of known asbestos structure concentrations, reference filters shall be examined. Reference filters should be maintained as part of the laboratory's Quality Assurance program.

5. To minimize subjective effects, some specimens should be recounted by a different microscopist.

6. Asbestos laboratories shall be accredited by the National Voluntary Laboratory

Accreditation Program.

7. At this time, performance evaluation samples for asbestos in air are not available for Removal Program Activities.

9.2 PCM Requirements

1. Examine reference slides of known concentration to determine the analyst's ability to satisfactorily count fibers. Reference slides should be maintained as part of the laboratory's quality assurance program.

2. Examine field blanks to determine if there is contamination by extraneous structures during sample handling.

3. Some samples should be relabeled then submitted for counting by the same analyst to determine possible bias by the analyst.

4. Participation in a proficiency testing program such as the AIHA-NIOSH proficiency analytical testing (PAT) program.

## 10.0 DATA VALIDATION

Results of quality control samples will be evaluated for contamination. This information will be utilized to qualify the environmental sample results accordingly with the project's data quality objectives.

## 11.0 HEALTH AND SAFETY

When working with potentially hazardous materials, follow U.S. EPA, OSHA, and corporate health and safety procedures. More specifically, when entering an unknown situation involving asbestos, a powered air purifying respirator (PAPR) (full face-piece) is necessary in conjunction with HEPA filter cartridges. See applicable regulations for action level, PEL, TLV, etc. If previous sampling indicates asbestos concentrations are below personal health and safety levels, then Level D personal protection is adequate.

## 12.0 REFERENCES

1. Environmental Asbestos Assessment Manual, Superfund Method for the Determination of Asbestos in Ambient Air, Part 1: Method, EPA/540/2-90/005a, May 1990, and Part 2: Technical Background Document, EPA/540/2-90/005b, May 1990.

2. Methodology for the Measurement of Airborne Asbestos by Electron Microscopy, EPA's Report No. 68-02-3266, 1984, G. Yamate, S.C. Agarwal, and R. D. Gibbons.

3. National Institute for Occupational Safety and Health. NIOSH Manual of Analytical Method. Third Edition. 1987.

4. U.S. Environmental Protection Agency. Code of Federal Regulations 40 CFR 763. July 1, 1987. Code of Federal Regulations 40 CFR 763 Addendum. October 30, 1987.

5. U.S. Environmental Protection Agency. Asbestos-Containing Materials in Schools; Final Rule and Notice. 52 FR 41826.

6. Occupational Safety and Health Administration. Code of Federal Regulations 29 CFR 1910.1001. Washington, D.C. 1987.

APPENDIX A
Tables
SOP #2015
November 1994

TABLE 1.

SAMPLE STATIONS FOR OUTDOOR SAMPLING

| Sample Station Location | Sample Numbers | Rationale |
|---|---|---|
| Upwind/Background[(1)] | Collect a minimum of two simultaneous upwind/background samples 30$^{o}$ apart from the prevailing windlines. | Establishes background fiber levels. |
| Downwind | Deploy a minimum of 3 sampling stations in a 180 degree arc downwind from the source. | Indicates if asbestos is leaving the site. |
| Site Representative and/or Worst Case | Obtain one site representative sample which shows average condition on-site or obtain worst case sample (optional). | Verify and continually confirm and document selection of proper levels of worker protection. |

(1) More than one background station may be required if the asbestos originates from different sources.

TABLE 2

SAMPLE STATIONS FOR INDOOR SAMPLING

| Sample Station Location | Sample Numbers | Rationale |
|---|---|---|
| Indoor Sampling | If a work site is a single room, disperse 5 samplers throughout the room.<br><br>If the work site contains up to 5 rooms, place at least one sampler in each room.<br><br>If the work site contains more than 5 rooms, select a representative sample of the rooms. | Establishes representative samples from a homogeneous area. |
| Upwind/Background | If outside sources are suspected, deploy a minimum of two simultaneous upwind/background samples 30$^{o}$ apart from the prevailing windlines. | Establish whether indoor asbestos concentrations are coming from an outside source. |
| Worst Case | Obtain one worst case sample, i.e., aggressive sampling (optional). | Verify and continually confirm and document selection of proper levels of worker protection. |

APPENDIX B
Figures
SOP #2015
November 1994

FIGURE 1. Transmission Electron Microscopy Filter Cassette

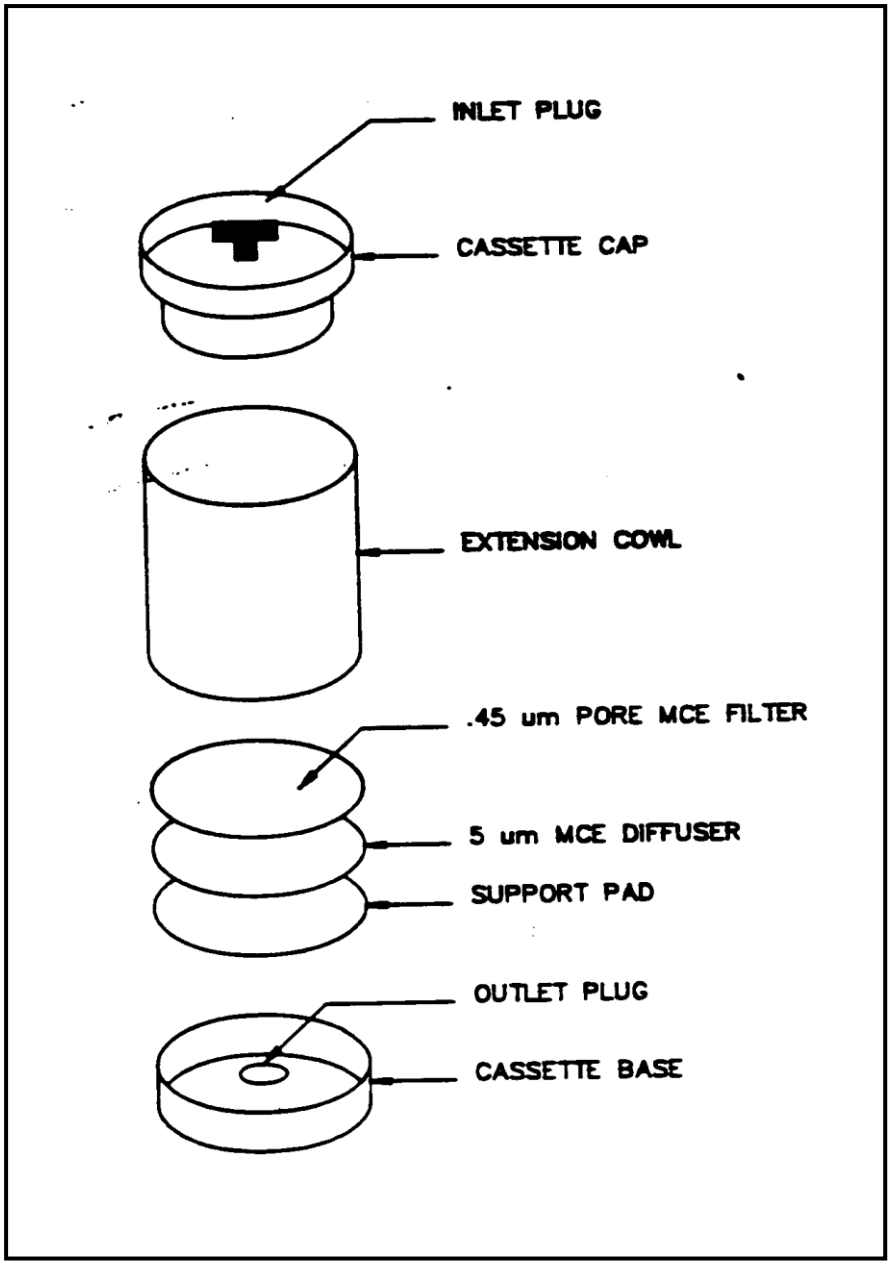


FIGURE 2. Phase Contrast Microscopy Filter Cassette

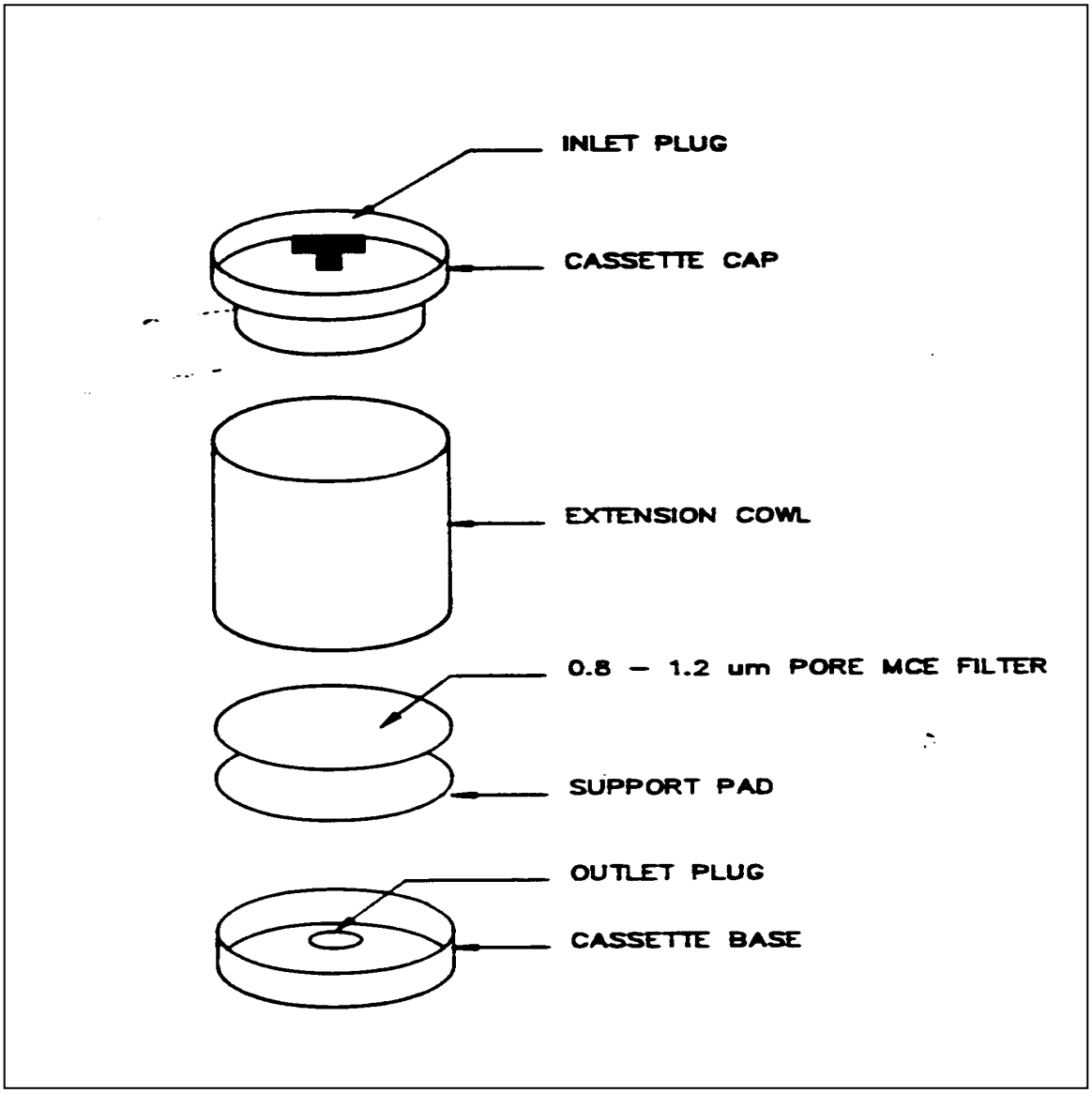


FIGURE 3. Calibrating a Personal Sampling Pump with a Bubble Meter

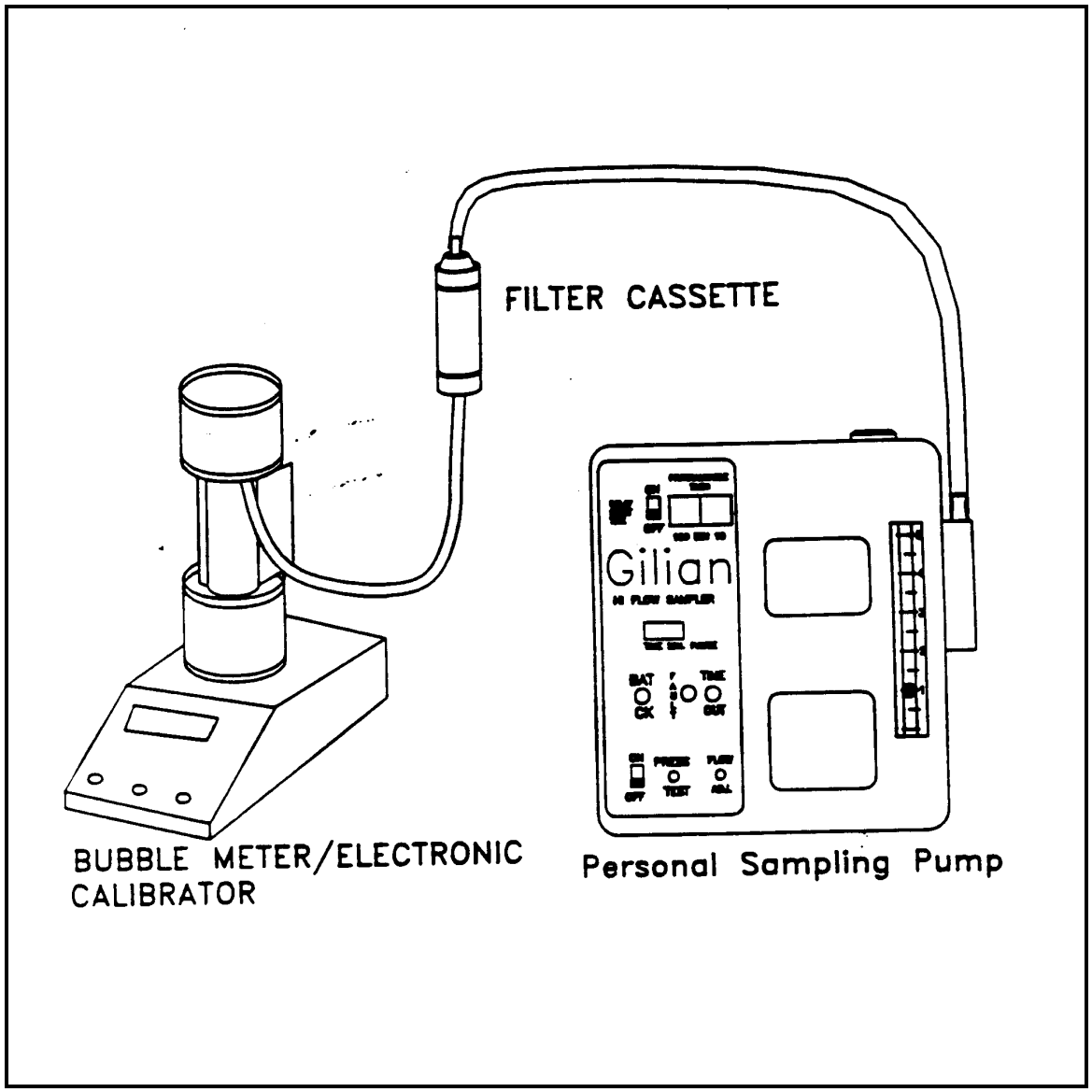


FIGURE 4. Calibrating a Rotameter with a Bubble Meter

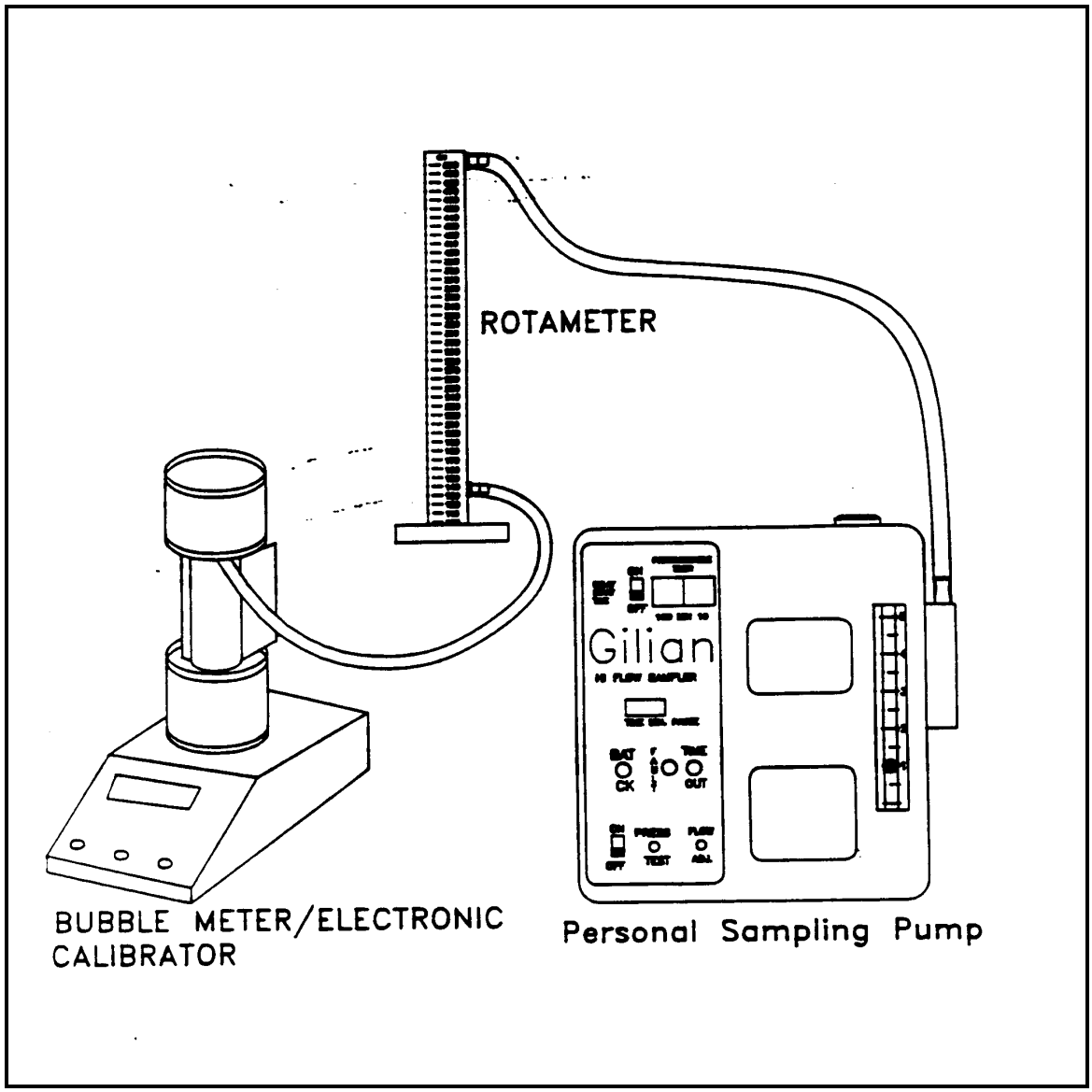


FIGURE 5. Calibrating a Sampling Pump with a Rotameter

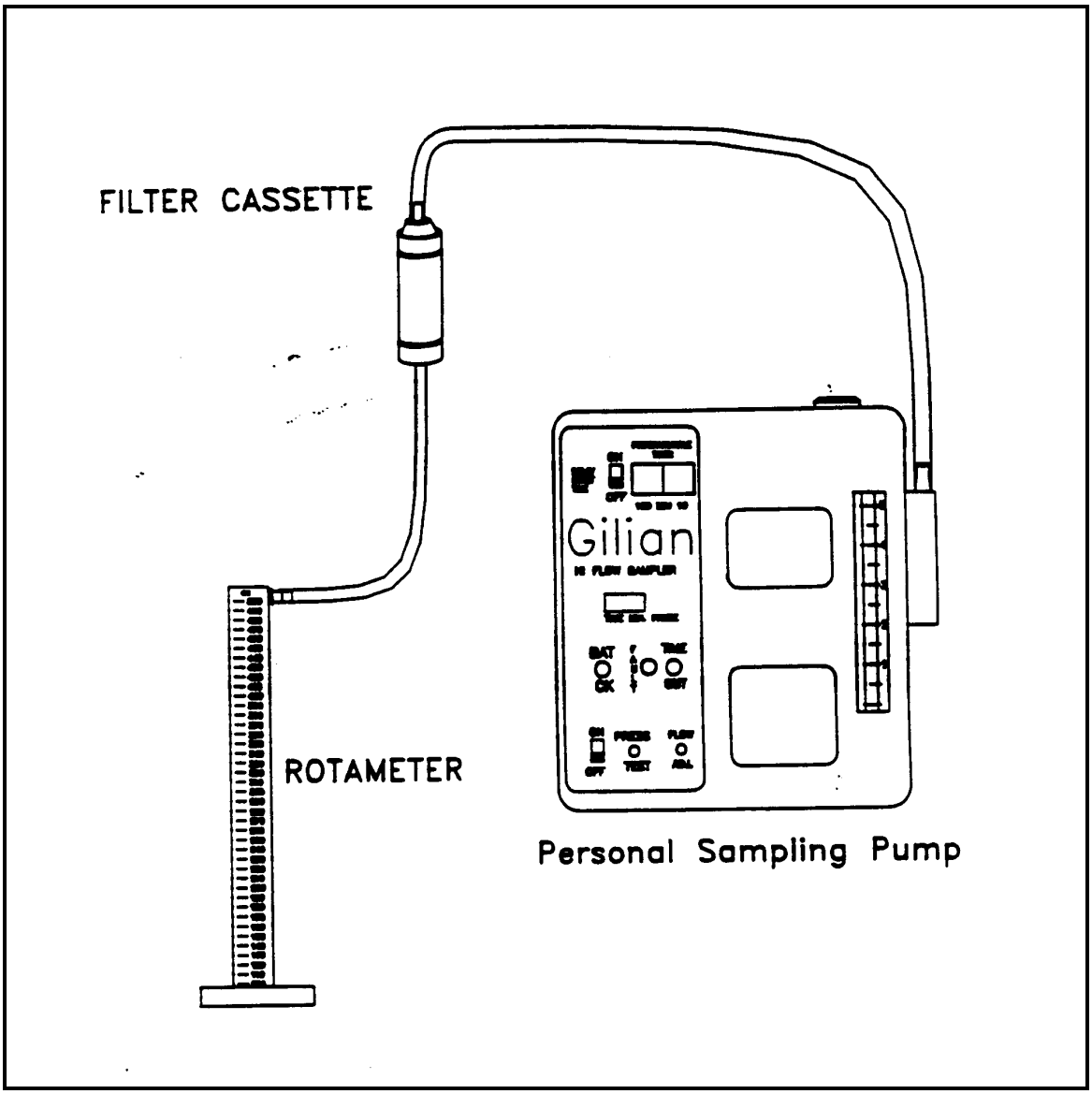


FIGURE 6. Personal Sampling Train for Asbestos

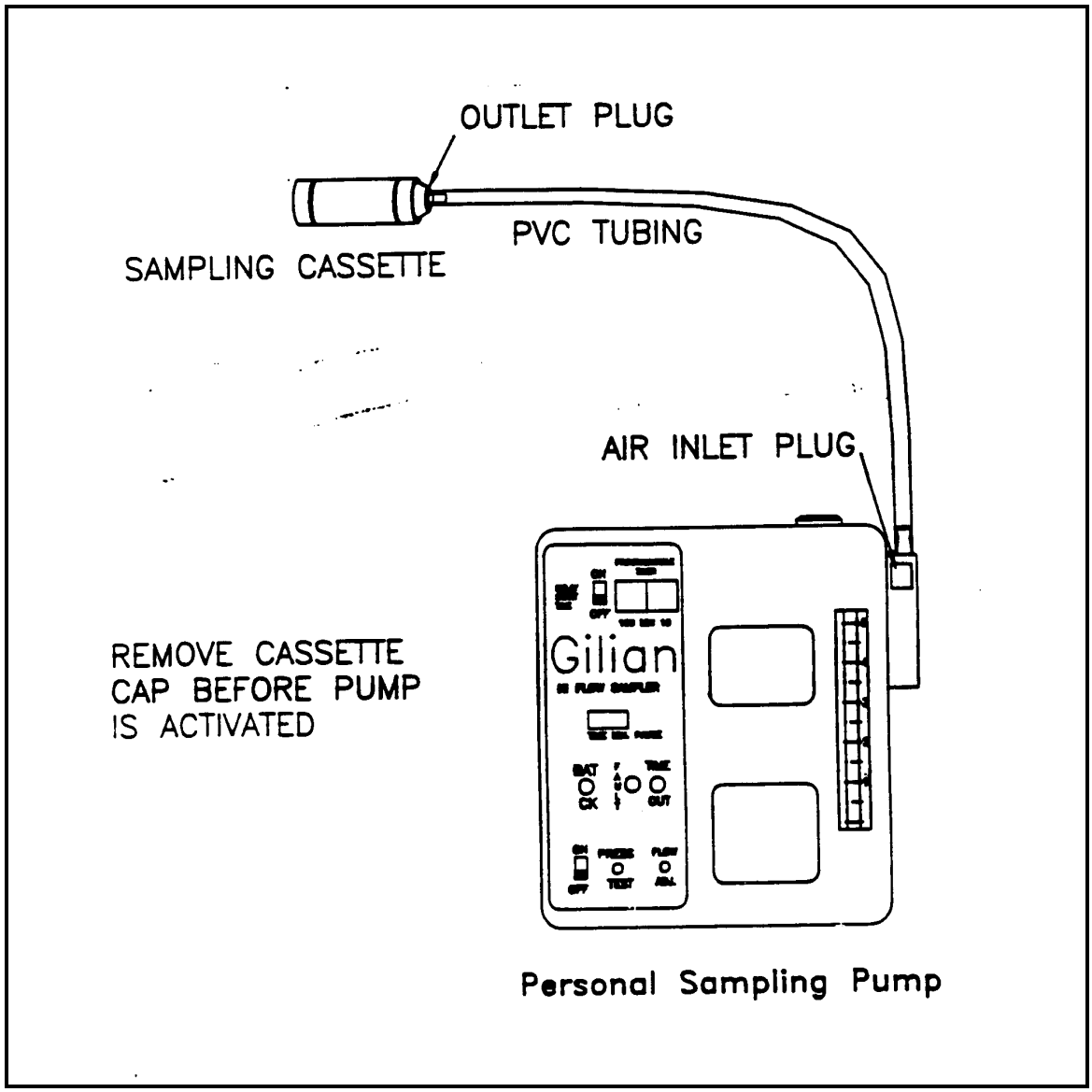
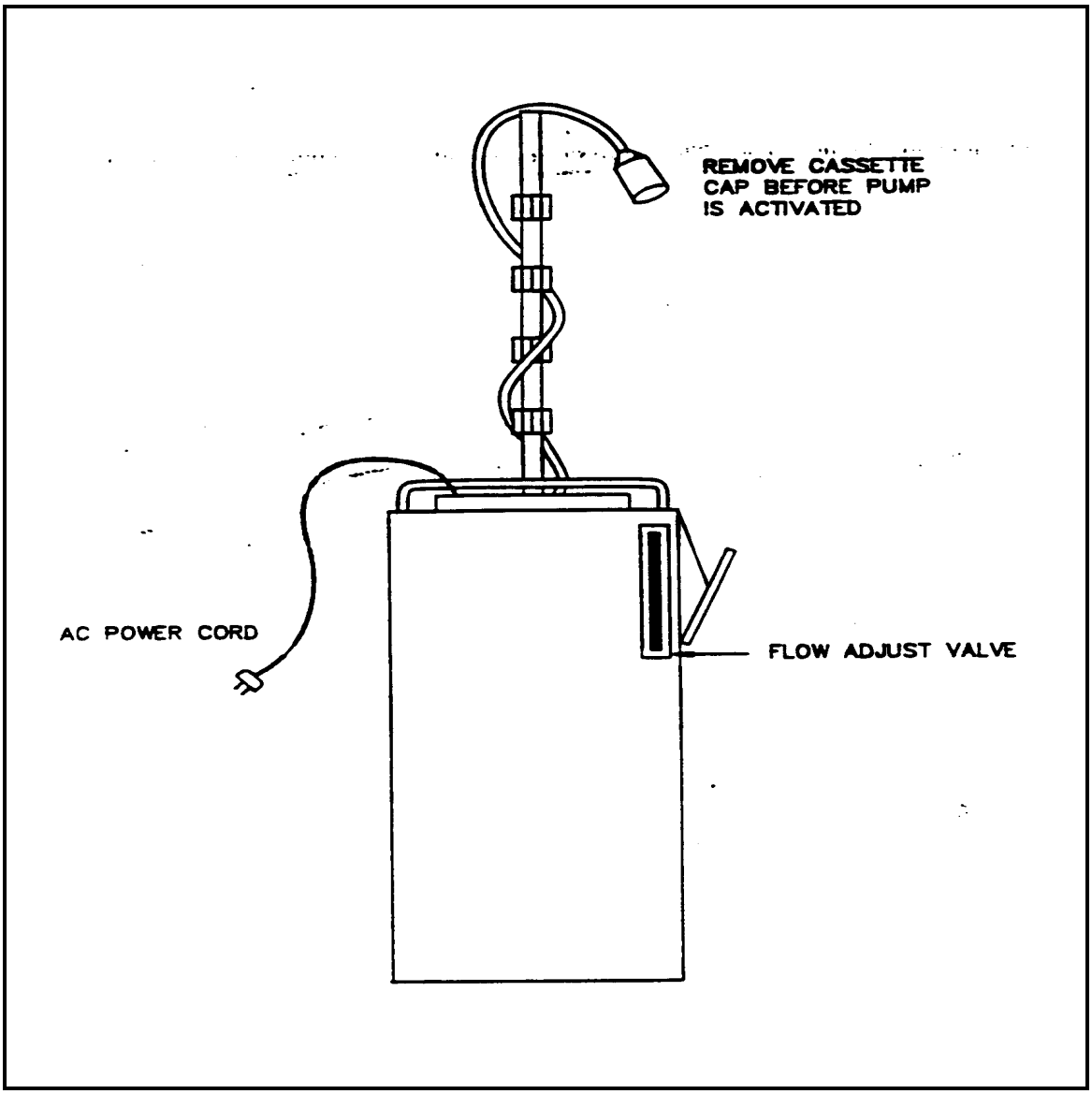


FIGURE 7. High Flow Sampling Train for Asbestos



Response Engineering and Analytical Contract

# STANDARD OPERATING PROCEDURES



**DATA VERIFICATION/VALIDATION PROCEDURE FOR ASBESTOS IN AIR BY TEM (BASED ON ISO 10312)**

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CONTENTS

CONTENTS (cont'd)

**DATA VERIFICATION/VALIDATION PROCEDURE FOR ASBESTOS IN AIR BY TEM (BASED ON ISO 10312)**

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CONTENTS (cont'd)

**DATA VERIFICATION/VALIDATION PROCEDURE FOR ASBESTOS IN AIR BY TEM (BASED ON ISO 10312)**

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## 1.0 OBJECTIVE

The objective of this Standard Operating Procedure (SOP) is to establish protocols for the evaluation, verification and validation of asbestos samples in air by transmission electron microscopy (TEM). These data verification/validation procedures are based on the analytical procedures in the International Organization for Standardization (ISO) Method 10312:1995 and the National Environmental Laboratory Accreditation Committee (NELAC) Interim Standard for Asbestos Testing.

## 2.0 APPLICABILITY

This SOP is applicable to asbestos air samples analyzed by TEM and submitted to the Response Engineering and Analytical Contract (REAC) Data Validation and Report Writing (DV&RW) Group for data verification/validation as required by the project's data quality objectives (DQOs).

## 3.0 DESCRIPTION

All required data reduction, reporting and documentation submitted by the laboratory will be used to assess the validity and accuracy of the data package. Asbestos results received from the laboratory may include hard copy final results, laboratory logbooks and electronic deliverables such as the National Asbestos Data Entry Spreadsheets (NADES) in Excel and Portable Document Format (pdf) final reports. The NADES Excel files (Data Entry 1, Data Entry 2 and NADES tabs) are verified by comparing Chain of Custody data (i.e., sample number, sample volume, date of sampling, etc.) and laboratory handwritten log sheets (grid openings, total number of structures, error codes, etc.) to identify discrepancies that may impact the Certificate of Analysis reported by the laboratory. An in-house program (CheckNades) is used to compare the information in the NADES with data in the sample SCRIBE database file (i.e., sample volumes, missing data, missing sample numbers, and incorrect sample numbers). The Scribe information should be verified against the field data sheets. Results are then corrected for volume and sample number discrepancies, checked for error codes and then summarized in a validation report. The laboratory will be contacted to make any necessary corrections and reissue any corrected NADES or laboratory certificates of analysis. When the laboratory makes changes to the NADES spreadsheet, they should be documenting the revision by picking the appropriate correction number in cell M1 on the Data Entry 1 tab in NADES.

### 3.1 Data Validation Qualifiers

The following qualifiers will be assigned to results during the data review process. A definition of each of the qualifiers is listed in the following table.

| | |
|---|---|
| U | The analyte was analyzed for, but was not detected above the analytical sensitivity |
| J | The result is an estimated quantity. The associated numerical value is the approximate concentration of the analyte in the sample. |
| R | The data are unusable. The sample results are rejected due to serious deficiencies in meeting Quality Control (QC) criteria. The analyte may or may not be present in the sample. |
| UJ | The analyte was analyzed for, but was not detected. The reporting limit is approximate and may be inaccurate or imprecise. |

3.2 Selection of Samples and Check of NADES Excel Files

3.2.1 Objective

The information in the NADES Excel files will be compared against the information in the Scribe database file. To facilitate this process, an in-house developed CheckNades program will be used to flag any anomalies relating to sample numbers, flow volumes and data entry errors.

3.2.2 Requirements

A text file (.txt) is generated using the CheckNades program. Windows XP operating system, Microsoft Office 2003 and the latest version of CheckNades must be installed on each local computer. A copy of the check NADES report should be included in the data validation report.

3.2.3 Evaluation Procedure

3.2.3.1 Check the resulting .txt file for any data entry error codes.

3.2.3.2 Check the .txt file for discrepancies in flow volumes or sample numbers. Resolve and rectify discrepancies using other supporting documentation such as logbooks, sample labels or air sampling worksheets.

3.2.3.3 Select 10 percent (%) of the samples with asbestos counts using a random number generator. If the total number of samples with reportable concentrations is less than the required 10%, randomly select the additional samples from the remaining samples with no reportable concentrations.

3.2.4 Action

List any error codes and the possible reason(s) for the errors on the data assessment form (Appendix A, Figure 1). List any sample number, sample volume or missing sample information discrepancies on the data assessment form. Contact the laboratory to correct these discrepancies and reissue the data package.

3.3 Asbestos Identification and Quantification

3.3.1 Objective

To ensure the proper quantification of asbestos structures, the minimum number of grid openings required to achieve a particular analytical sensitivity and limit of detection must be verified. If the grid exhibits more than approximately 10% obscuration in the majority of the grid openings, the grid is overloaded. Data Quality Objectives for some projects may permit a loading of up to 25%. However, this determination is a variation from the standard method and must be made on a case by case basis. Verification of the types of asbestos structures reported by the laboratory with the exception of the information on the laboratory bench sheets is outside the scope of this SOP.

3.3.2 Requirements

The information from the laboratory's bench sheets (grid identification, grid opening, structure type, number of primary and secondary structures, length and width dimensions, identification code and mineral type) must be transcribed correctly to the NADES (Data Entry 2 Tab).

The following information ( magnification, date received, lab sample number, COC number, field or QC sample type, media, volume, # grids prepared, filter area in square millimeters ($mm^2$) and analysis date) must be transcribed correctly to Data Entry 1 Tab of the Excel file for selected files.

The totals on the NADES Spreadsheet (NADES Report Tab) for the various types of structures (i.e., counting rules) must be confirmed for the selected samples. This may include total EPA Structures, PCME structures, AHERA structures and/or Berman Crump structures, depending on the project's data

quality objectives. Sensitivity, density, concentration and reporting limits must be verified for each of the selected samples.

3.3.3 Evaluation Procedure

Determine if there are any site-specific requirements for analytical sensitivity or limit of detection.

Verify that the calculations for analytical sensitivity, concentrations, density and reporting limit (RL) have been done correctly using the following equations:

Analytical Sensitivity, S/cc = (1 structure x EA) / (1000 x #GO x V x GOA)

Concentration, S/cc = Analytical Sensitivity x Total # Structures

Density, $S/mm^2$ = V x 1000 x Concentration/EA

RL, $S/mm^2$ =Density/Total # Structures

Where:

S/cc = Structures/cubic centimeter
EA = Effective filter area, $mm^2$
GOA = Grid opening area, $mm^2$
V = sample volume, in liters (L)
RL = Reporting limit, $S/mm^2$
#GO = grid openings

Verify that the correct number of grid openings was used to achieve the analytical sensitivity for the method and/or project. Refer to Table 1 in ISO10312 (make sure units are reported in the same units).

Verify that the information from the laboratory's bench sheets (grid identification, grid opening, structure type, number of primary and secondary structures, length and width dimensions, identification code and mineral type) have been transcribed correctly to the Data Entry 2 Tab of the NADES.

Verify the following information on the Data Entry 1 Tab of the Excel file for selected files: magnification, date received, lab sample number, COC number,

field or QC sample type, media, volume, # grids prepared, filter area in square millimeters ($mm^2$) and analysis date.

Verify the totals on the NADES Report Tab for the various types of structures (i.e., counting rules) for the selected samples. This may include total EPA `Structures, PCME structures, AHERA structures and/or Berman Crump structures, depending on the project's DQOs. Use the equations below to check sensitivity, density, concentration and reporting limits. Verify the analytical sensitivity limits using the project-defined data quality objectives.

3.3.4 Action

List any discrepancies in the validation report. If errors are found, return the data package to the laboratory and request that a corrected data package be submitted. The revised data from the lab shall subsequently be validated per this SOP to ensure that all anomalies/discrepancies have been addressed.

Document any intentional deviations from the method in the validation report.

Note any grid overload in the validation report.

3.4 Blanks

3.4.1 Objective

The results of the blank analyses are assessed to determine the existence and magnitude of contamination problems. If any problems with the blanks exist, all data associated with the batch must be carefully evaluated to determine whether or not there is an inherent contamination for the samples in the batch, or the problem is an isolated occurrence not affecting the sample data.

3.4.2 Requirements

3.4.2.1 Lot or Batch (Laboratory) Blank

A blank filter shall be prepared with each set of samples. A blank filter is left uncovered during the preparation of a sample set and is prepared alongside the samples (this is conducted by the laboratory). At least one laboratory blank is prepared with each batch of samples with the frequency of one in 25 samples. NELAC Volume 1, Module 3 states that the maximum contamination per filter shall be no more than 53 structures/$mm^2$ with a maximum average contamination for all blank filters not to exceed 18 structures/$mm^2$.

**NOTE:** According to NELAC policies and procedures, a laboratory's SOP and/or method takes precedence over the NELAC requirements. The data quality objectives for a specific project takes precedence over all of the above (e.g., one lot blank per day of sampling with an acceptance criterion of <10 structures/mm$^2$).

3.4.2.2 Field Blank

ISO10312 states that at least one field blank shall be processed along with each batch of samples. There is no field blank requirement in the NELAC policies and procedures; however, the criteria listed above for the lot or batch blank will be used if there are no criteria listed in an approved Quality Assurance Project Plan (QAPP) for a specific project takes precedence over all of the above (e.g., one field blank per day with an acceptance criterion of <20 structures/mm$^2$).

3.4.3 Evaluation Procedure

Review the blanks reported on the results summary (laboratory certificate of analysis) and on the NADES with the blank results on the laboratory bench sheets. Verify that the results were correctly transcribed into the NADES.

Verify that a lot or batch blank was done at the frequency of one in 25 samples.

Verify that at least one field blank was processed along with each batch of samples.

Verify that the maximum contamination per filter shall be no more than 53 structures/mm$^2$ with a maximum average contamination for all blank filters not to exceed 18 structures/mm$^2$. Alternatively, verify that the contamination per filter does not exceed the project's DQOs.

3.4.4 Action

Qualify the data based on the following table.

| Blank Result | Sample Result | Action for Samples |
|---|---|---|
| Frequency < 4% | | Use professional judgment |
| >RL | > RL > Blank | Use professional judgment or qualify sample results as estimated |
| >RL | ≤ RL | Report RL with a "U" |
| >RL | ≥10x Blank Result | No Action |

3.5 Analytical Variability

3.5.1 Objective

Duplicate/replicate sample analyses are used to demonstrate acceptable method precision by the laboratory at the time of analysis.

3.5.2 Requirements

Analytical variability is determined using recounts between and within microscopists and between laboratories. Recounts of 10% of specimens are necessary to minimize subjective effects in the reported results. Repeat results should not differ at the 5% significance level (defined as the mean plus or minus 2 times the standard deviation).

NELAC (Volume 1, Module 3) requires that replicate, duplicate and verified analyses must be conducted by the laboratory.

3.5.2.1 Replicate Analysis

A replicate performed by a second independent analyst on the same grids but different grid openings must be done at a frequency of one in 100 samples with results within 1.5x of Poisson standard deviation.

3.5.2.2 Duplicate Analysis

A duplicate, using a second wedge from the sample filter prepared and analyzed in the same manner as the original samples, must be done at a frequency of one in 100 samples with results within 2x of Poisson standard deviation, analyzed by the same analyst.

3.5.2.3 Verified Analysis

A second independent analysis on the same grids and same grid openings used for the original analysis must be performed at the frequency of one in 20 samples.

Generally samples identified as field blanks are not used for the duplicate analysis.

3.5.3 Evaluation Procedure

Verify that the replicate, duplicate and verified analyses were done as in Sections 3.5.2.1 through 3.5.2.3.

Verify whether the results fall within established control limits.

Check the raw data to verify that results have been correctly transcribed to the NADES.

Verify that at least two grid preparations and four grid openings be examined with at least one grid opening from each of the two preparations.

3.5.4 Action

Using the information provided by the laboratory and the precision criteria in the laboratory's SOP, qualify the sample used for precision estimated (J) if outside the laboratory's acceptance criterion.

3.6 Equipment Performance Checks

3.6.1 Objective

Equipment performance checks ensure that the TEM is capable of producing acceptable quantitative data. Calibration of the TEM screen magnification, camera constant and Energy Dispersive X-Ray Analysis (EDXA) system assure that adequate quantitative and qualitative results are attainable from the system. All calibrations must be performed under the same analytical conditions as that used for analysis. The inability of a laboratory to perform acceptably indicates that severe problems may exist in the analytical system. Any data generated under such conditions should be considered suspect. If improper calibration procedures were used, all data associated with that calibration should be re-analyzed. If the data in question are needed on a priority basis, professional judgment may be applied to determine to what extent the data may be utilized.

3.6.2 Requirements

3.6.2.1 Calibration of TEM Screen Magnification

On a monthly basis, the electron microscope must be calibrated at the fluorescent screen at the magnification typically used for counting (i.e., 15,000 to 20,000x).

3.6.2.2 Camera Constant Calibration

When used in the Energy Dispersion (ED) mode, the camera constant of the TEM must be calibrated. NELAC recommends monthly to establish the stability of the camera constant. A criterion of <10% variation is used for the medium used for routine measurements (e.g., screen or film). A criterion of <5% is used for patterns on permanent media

3.6.2.3 EDXA System Calibration

ISO 10312 specifies that an energy calibration of the EDXA system for a low energy and a high energy peak be performed on a regular basis. NELAC states that initially and/or prior to analysis, the EDXA system must be calibrated to within 20 eV for at least 2 peaks between 0.7 keV and 10 keV (one between 0.7 - 2 keV and one between 7 - 10 keV).

3.6.2.4 k-Factors

ISO 10312 specifies the use of reference silicate minerals to calculate k -factors. The relationship between Mg, Al, K, Ca, Mn and Fe to Si must be calculated for each of the elements typically found in asbestos. For a particular instrument and particle size, the value of k should remain constant. NELAC states that the k-factors be calculated semi-annually or anytime the detector geometry has been altered using National Institute of Standards and Technology (NIST) Standard

Standard Reference Materials (SRMs) or other appropriate certified materials. The k-factors shall be determined to a precision (2s) within 10% relative to the mean value obtained for Mg, Al, Si, Ca and Fe, and within 20% relative to the mean for Na. Acceptance criteria are as follows:

| K-Factor | PASS Criteria | Acceptance Criteria |
|---|---|---|
| Mg-Si | 1.0 - 2.0 | 2s < 10% Mean |
| Ca-Si | 1.0 - 1.75 | 2s < 10% Mean |
| Fe-Si | 1.0 -2.0 | 2s < 10% Mean |
| Mg-Fe | < 1.5 | N/A |
| Na-Si | 1.0 - 4.0 | 2s < 20% Mean |
| Al-Si | 1.0 - 1.75 | 2s < 10% Mean |

3.6.3 Evaluation Procedure

Verify that the laboratory has calibrated the electron microscope at the fluorescent screen at the magnification typically used for counting (i.e., 15,000 to 20,000x) using copies of logbook pages and/or calibration data displayed on control charts that show trends over time. Consult the laboratory's SOP for frequency and acceptance criterion.

Verify that the ED camera constant has been calculated for both the screen and photographic plate or film using copies of logbook pages and/or calibration data displayed on control charts that show trends over time should be submitted. Consult the laboratory's SOP for frequency and acceptance criterion.

Verify that the laboratory has calibrated the EDXA system prior to analysis and the system was recalibrated if outside the specified range.

Verify that the k-factors have been calculated on a semiannual basis and are within the acceptance criteria listed above in the requirements section.

3.6.4 Action

If the TEM screen magnification has not been conducted at the required frequency or is not within the acceptance criterion, qualify the data estimated (J).

If the camera constant has not been performed with the frequency specified or the variability exceeds acceptance criterion, qualify the data estimated (J).

If the calibration of the EDXA system failed and was not recalibrated, qualify the data unusable (R).

If k-factors are not available in the data package, qualify data estimated (J).

| Calibration Result | Action for Samples |
|---|---|
| Calibration not performed or failed | Qualify all results unusable (R) |
| Calibration not performed at the required frequency | Use professional judgment<br>Qualify results ≥RL estimated (J)<br>Qualify non-detects estimated (UJ) |
| Variability exceeds acceptance criterion | Qualify results ≥RL estimated (J)<br>Qualify non-detects estimated (UJ) |

3.7 Additional System Checks

3.7.1 Objective

Additional system checks assure that the X-ray beam stability and detector resolution are within criteria. These additional checks will be monitored using the calibration reports (tables and/or charts) supplied by the laboratory.

3.7.2 Requirements

3.7.2.1 Beam Dose Check

The beam dose must be calibrated so that beam damage to chrysotile is minimized, specifically so that an electron diffraction pattern from a single fibril greater than 1 micron (μm) in length from a NIST SRM chrysotile sample is stable in the electron beam dose for at least 15 seconds.

3.7.2.2 Spot Size Check

The diameter of the smallest beam spot at crossover must be less than 250 μm and calibrated quarterly.

3.7.2.3 Detector Resolution Check

The detector resolution must be checked quarterly to ensure a full-width half maximum resolution of less than 175 electron volts (eV) at Mn Ka (5.90 eV).

3.7.2.4 Detector Sensitivity

The sensitivity of the detector must be documented quarterly by collecting resolvable magnesium (Mg) and silicon (Si) peaks from a unit fibril of NIST SRM 1866 chrysotile.

3.7.3 Evaluation Procedure

Verify that at least 90% of the pattern is stable for 15 seconds. Verify that this was conducted on a monthly basis.

Verify that the diameter of the smallest beam spot at crossover is less than 250nm on a quarterly basis.

Verify that the detector resolution was checked quarterly and is less than 175 nm.

Verify that the laboratory has analyzed the appropriate NIST SRM on a quarterly basis.

3.7.4 Action

If provided by the laboratory, verify the calibration reports (tables and/or charts) which are generated as part of an on-going performance monitoring program. Alternatively, a statement to the fact in the laboratory case narrative that the ISO Method and/or NELAC requirements are met is sufficient.

4.0 RESPONSIBILITIES

4.1 QA/QC Chemist

The QA/QC Chemist must have a working knowledge of the method used to obtain the data and must ensure that all documents included in the data package are complete. The QA/QC Chemist is responsible for informing the Data Validation and Report Writing (DV&RW) Group Leader of any major deviations from the method that may affect the usability of the data.

The QA/QC Chemist will prepare data assessment forms and a written validation report of any anomalies. The QA/QC Chemist will prepare written communication to the laboratories detailing any deficiencies and will request that the laboratory correct any errors and resubmit the data.

The QA/QC Chemist is also responsible for generating nonconformance memos due to a nonconformance or deficiency in the method. These nonconformance memos will be submitted to the Quality Assurance Officer (QAO) for verification.

4.2 Data Validation and Report Writing Group Leader

The DV&RW Group Leader is responsible for updating this SOP as requirements change. All changes must be documented on a SOP Change Form and approved by the QAO prior to implementation.

The DV&RW Group Leader is responsible for assigning validation priorities based on the due date of a project and for the timely delivery of the validation report to the client within two weeks of data package receipt. The DV&RW Group Leader periodically audits the review process to ensure compliance with review requirements.

The DV&RW Group Leader is responsible for communication of any major noncompliance of the method that may affect the usability of the data to the REAC Task Leader and QAO.

The DV&RW Group Leader ensures that all data review, verification and validation documentation is complete for each project and is archived in accordance with standard REAC archival procedures.

4.3 Quality Assurance Officer

The QAO is responsible for providing oversight to the DV&RW Group by providing technical guidance, ensuring adherence to this SOP and conducting routine audits of the data validation process.

The QAO is responsible for verifying any nonconformance memos generated by the data review, verification and validation process.

5.0 APPENDICES

A - Asbestos in Air by TEM Data Assessment Form

**DATA VERIFICATION/VALIDATION PROCEDURE FOR ASBESTOS IN AIR BY TEM (BASED ON ISO 10312)**

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REFERENCES

International Organization for Standardization (ISO). 1995. *Ambient air - Determination of asbestos fibres - Direct-transfer transmission electron microscopy method*, Method 10312:1995(E).

National Environmental Laboratory Accreditation Committee (NELAC). 2007. *Asbestos Testing Interim Standard*, Volume 1, Module 3, December 2007.

**DATA VERIFICATION/VALIDATION PROCEDURE FOR ASBESTOS IN AIR BY TEM (BASED ON ISO 10312)**

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APPENDIX A
Asbestos in Air by TEM Data Assessment Form
SOP #1025
February 2008

## DATA VERIFICATION/VALIDATION PROCEDURE FOR ASBESTOS IN AIR BY TEM (BASED ON ISO 10312)

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**Asbestos in Air by TEM (ISO 10312:1995) Data Assessment Form**

**PROJECT NAME _____WA#_____PACKAGE #_____**

**# OF SAMPLES _____ MATRIX _____CHAIN OF CUSTODY_____**

The data verification/validation procedures are based on the analytical procedures in the International Organization for Standardization (ISO) Method 10312:1995 and the National Environmental Laboratory Accreditation Committee (NELAC) Interim Standard for Asbestos Testing dated 12/28/2007. Professional judgment should be used for any anomalies not included in this data assessment form.

At present, asbestos results received from the laboratory may include hard copy final results, laboratory logbooks and electronic deliverables such as National Asbestos Data Entry Spreadsheets (NADES) in Excel and Portable Document Format (pdf) final reports. The NADES Excel files (Data Entry 1, Data Entry 2 and NADES) are verified by comparing Chain of Custody data (i.e., sample number, sample volume, date of sampling, etc.) and laboratory handwritten log sheets (grid openings, total number of structures, error codes, etc.) to identify discrepancies that may impact the Certificate of Analysis reported by the laboratory. To facilitate this task, an in-house program (CheckNades) is used to compare the information in the NADES with the data in the SCRIBE database file (i.e., sample volumes, missing data, missing sample numbers, incorrect sample numbers). The Scribe entries should be verified against the field data sheets. Results are then corrected for volume and sample number discrepancies, checked for error codes and then summarized in a validation report. The laboratory will be contacted to make any necessary corrections and reissue any corrected NADES or laboratory certificates of analysis.

At a minimum, ten percent of the total samples submitted on the Chain of Custody (COC) that indicate the presence of mineral structures/asbestos are randomly selected using a random number generator for validation. This validation entails the verification of analytical sensitivity, density, concentration and reporting limits on the selected samples. Additional QC samples, lot or batch blanks, field blanks, duplicate sample analysis (different preparation by same analyst), replicate analysis (same preparation by a different analyst), reference standards (e.g., NIST SRM 1876b) and equipment performance checks, as provided, are reviewed for compliance to the method and/or the laboratory's standard operating procedure (SOP). The minimum of ten percent of the samples are then checked for deviations from the analytical method and laboratory sub-contract Statement of Work (SOW). The Data Validation and Report Writing Group Leader shall be informed of any deviations from the analytical methodology. A nonconformance memo will be generated for any deviations. This information will be included in the validation report.

**NOTE:** This data assessment form includes the quality control (QC) requirements listed in ISO 10312, Annex B of ISO 10312 and NELAC. Data assessment for each specific project may vary based on the information supplied by the laboratory. If the laboratory does not provide any portion of the information listed on pages 7 to 9 of this data assessment form, a statement by the laboratory in the case narrative documenting that the ISO Method and/or NELAC requirements have been met will be accepted in lieu of the supporting documentation. This statement will be included in the validation report and included as an appendix for documentation.

Validator's Signature: _____ Date: _____/_____/_____

**DATA VERIFICATION/VALIDATION PROCEDURE FOR ASBESTOS IN AIR BY TEM (BASED ON ISO 10312)**

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**1. Selection of Samples and Check of NADES Excel Files**

Once the laboratory results files are received in Excel format, the CheckNades program will be run using the project-specific Scribe file and the Excel results files. Each folder that contains laboratory results must be run against the Scribe .mdb file. A text (.txt) file will be generated that will detect any anomalies relating to error codes or flow volumes.

A) Check the resulting .txt file for error codes. List the error codes and the possible reason(s) for the errors below and tabulated in the validation report.

B) Check the resulting .txt file for any discrepancies in flow volumes or sample numbers between the Scribe information and information entered by the laboratory. Use supporting documentation such as field sampling worksheets or sample labels to confirm which entry is correct. List any discrepancies below and in the validation report.

C) Select 10% of the samples with counts using a random number generator (e.g., in Excel). If the total number of samples with reportable concentrations is less than the required 10%, randomly select additional samples from the remaining samples with no counts. List the selected samples below.

**3. Laboratory Blanks (Section 9.7 of ISO 10312)**

**Lot or Batch Blank**

A blank filter shall be prepared with each set of samples. A blank filter is left uncovered during the preparation of a sample set and is prepared alongside the samples (this is conducted by the laboratory). Verify that at least one laboratory blank is prepared with each batch of samples. Verify that the laboratory blanks are analyzed with the frequency of one in 25 samples. NELAC Volume 1, Module 3 states that the maximum contamination per filter shall be no more than 53 structures/$mm^2$ with a maximum average contamination for all blank filters not to exceed 18 structures/$mm^2$. Verify that the laboratory blanks meet these criteria. Note any discrepancies in the validation report.

**NOTE:** According to NELAC policies and procedures, a laboratory's SOP and/or method takes precedence over the NELAC requirements. The data quality objectives for a specific project takes precedence over all of the above (e.g., one lot blank per day of sampling with an acceptance criterion of <10 structures/$mm^2$).

A) Lot/Batch Blank contamination

**Field Blank**

ISO10312 states that at least one field blank shall be processed along with each batch of samples. There is no field blank requirement in the NELAC policies and procedures; however, the criteria listed above for the lot or batch blank will be used if there are no criteria listed in an approved Quality Assurance Project Plan (QAPP) for a specific project takes precedence over all of the above (e.g., one field blank per day with an acceptance criterion of <20 structures/$mm^2$).

B) Field Blank contamination

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**4. Analytical Variability (Section 9.7 of ISO 10312)**

Analytical variability is determined using recounts between and within microscopists and between laboratories. Recounts of 10% of specimens are necessary to minimize subjective effects in the reported results. Repeat results should not differ at the 5% significance level (defined as the mean plus or minus 2 times the standard deviation).

NELAC (Volume 1, Module 3) requires that replicate, duplicate and verified analyses are conducted as follows:

A) Replicate, by a second independent analyst on the same grids but different grid openings, at frequency of one in 100 samples, with results within 1.5x of Poisson standard deviation

B) Duplicate, using a second wedge from a sample filter prepared and analyzed in the same manner as the original samples, at a frequency of one in 100 samples, with results within 2x of Poisson standard deviation, analyzed by the same analyst.

C) Verified analysis, a second independent analysis on the same grids and same grid openings used for the original analysis, at a frequency of 1 in 20 samples.

Using the information provided by the laboratory and the precision criteria in the laboratory's SOP, qualify the sample used for precision estimated (J) if outside the laboratory's acceptance criterion. List any discrepancies below.

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**5. Equipment Performance Checks**

NOTE: If the laboratory does not provide any portion of the information listed on pages 7 to 9 of this data assessment form, a statement by the laboratory in the case narrative documenting that the ISO Method and/or NELAC requirements have been met will be accepted in lieu of the supporting documentation. This statement will be included in the validation report and included as an appendix for documentation.

**A) Calibration of TEM Screen Magnification**. On a monthly basis, the electron microscope must be calibrated at the fluorescent screen at the magnification typically used for counting (i.e., 15,000 to 20,000x).

Verify that the laboratory has performed this check. Copies of logbook pages with analyst's signature and date and/or calibration data displayed on control charts that show trends over time must be submitted. Consult the laboratory's SOP for frequency and acceptance criterion. If outside these limits, qualify data estimated (J).

**B) Camera Constant Calibration (both negative and on screen).** When used in the ED mode, the camera constant of the TEM must be calibrated. Verify that the ED camera constant has been calculated for both the screen and photographic plate or film. Copies of logbook pages with analyst's signature and date and/or calibration data displayed on control charts that show trends over time should be submitted. Consult the laboratory's SOP for frequency and acceptance criterion.

NOTE: NELAC recommends monthly to establish the stability of the camera constant. A criterion of <10% variation is used for the medium used for routine measurements (e.g., screen or film). A criterion of <5% is used for patterns on permanent media. If the camera constant has not been performed with the frequency specified or the variability exceeds acceptance criteria, data is qualified estimated (J).

**C) Calibration of EDXA System.** ISO 10312 specifies that an energy calibration of the EDXA system for a low energy and a high energy peak be performed on a regular basis. Copies of logbook pages with analyst's signature and date and/or calibration data displayed on control charts that show trends over time should be submitted. Consult the laboratory's SOP for frequency and acceptance criterion.

NOTE: NELAC states that initially and/or prior to analysis, the EDXA system must be calibrated to within 20 eV at least 2 peaks between 0.7 keV and 10 keV (one between 0.7 - 2 keV and one between 7 - 10 keV).

Verify that the laboratory has performed this check prior to analysis and the system was recalibrated if outside the specified range. If recalibration was not performed, data will be qualified unusable (R).

**D) k-Factors**. ISO 10312 specifies the use of reference silicate minerals to calculate k-factors. Verify that the relationship between Mg, Al, K, Ca, Mn and Fe to Si has been calculated for each of the elements typically found in asbestos. For a particular instrument and particle size, the value of k should remain constant. Copies of logbook pages with analyst's signature and date and/or calibration data displayed on control charts that show trends over time should be submitted. Consult the laboratory's SOP for frequency and acceptance criterion. If k-factors are not available in the data package, qualify data estimated (J).

NOTE: NELAC states that the k-factors be calculated semi-annually or anytime the detector geometry has been altered using National Institute of Standards and Technology (NIST) Standard Reference Materials (SRMs) or other appropriate certified materials. The k-factors shall be determined to a precision (2s) within 10% relative to the mean value obtained for Mg, Al, Si, Ca and Fe, and within 20% relative to the mean for Na. Acceptance criteria are as follows:

| K-Factor | PASS Criteria | Acceptance Criteria |
|---|---|---|
| Mg-Si | 1.0 - 2.0 | 2s < 10% Mean |
| Ca-Si | 1.0 - 1.75 | 2s < 10% Mean |
| Fe-Si | 1.0 -2.0 | 2s < 10% Mean |
| Mg-Fe | < 1.5 | N/A |
| Na-Si | 1.0 - 4.0 | 2s < 20% Mean |
| Al-Si | 1.0 - 1.75 | 2s < 10% Mean |

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**6. Additional System Checks**

Confirm that the laboratory has performed the equipment performance checks listed below. Copies of logbook pages with analyst's signature and date and/or calibration data displayed on control charts that show trends over time should be submitted in the data package. Consult the laboratory's SOP for frequency and acceptance criterion. Alternatively, a statement to the fact in the laboratory case narrative that the ISO Method and/or NELAC requirements are met is sufficient. If provided by the laboratory, verify the calibration reports (tables and/or charts), which are generated as part of an on-going performance monitoring program for the following:

A) Beam Dose Check - Verify that at least 90% of the pattern is stable for 15 seconds. NELAC recommends that this be conducted on a monthly basis.

B) Spot Size Check – Verify that the diameter of the smallest beam spot at crossover is less than 250 nanometers (nm) on a quarterly basis.

C) Detector Resolution Check – Verify that the detector resolution was checked quarterly and is less than 175 nm.

D) Resolvable Na, Mg and Si Peaks – Verify that the laboratory has analyzed the appropriate NIST SRM on a quarterly basis.